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A Summary of Current Program 7/1/62 and Preliminary Report of Progress for 7/1/60 to 6/30/62

EASTERN UTILIZATION RESEARCH AND DEVELOPMENT DIVISION
of the
AGRICULTURAL RESEARCH SERVICE
UNITED STATES DEPARTMENT OF AGRICULTURE

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This progress report of U.S.D.A. and cooperative research is primarily a tool for use of scientists and administrators in program coordination, development and evaluation; and for use of advisory committees in program review and development of recommendations for future research programs.

There is included under each problem area in the report a brief and very general statement on the nature of the research being conducted by the State Agricultural Experiment Stations and the professional manpower being devoted by the State stations to such research. Also included is a brief description of related work conducted by private organizations. No details on progress of State station or industry research are included except as such work is cooperative with U.S.D.A.

The summaries of progress on U.S.D.A. and cooperative research include some tentative results that have not been tested sufficiently to justify general release. Such findings, when adequately confirmed will be released promptly through established channels. Because of this, the report is not intended for publication and should not be referred to in literature citations. Copies are distributed only to members of Department staff, advisory committee members and others having a special interest in the development of public agricultural research programs.

This report also includes a list of publications reporting results of U.S.D.A. and cooperative research issued between July 1, 1960, and June 30, 1962. Current agricultural research findings are also published in the monthly U.S.D.A. publication, Agricultural Research. This progress report was compiled in the Eastern Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture, Philadelphia 18, Pa.

UNITED STATES DEPARTMENT OF AGRICULTURE
Washington, D. C.
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TABLE OF CONTENTS

INTRODUCTION

The mission of the Eastern Utilization Research and Development Division is the development of new and improved products and processes based on designated farm commodities, so as to create new and expanded markets for these commodities. The Division conducts research on dairy products, meat, animal fats, hides, tobacco, maple sirup, honey, and Eastern fruits and vegetables, including potatoes, and new crops.

In carrying out its mission, the Division does research in physical and biological science and engineering throughout the spectrum of basic research, applied research and pilot plant development. Division scientists are aware of the key role that basic research plays in uncovering new information that may be later exploited in applied research and development. Hence a substantial portion of the Division's effort is in basic research.

The Eastern Division has a total staff of about 390, including some 220 professional research scientists. The Division is organized in 10 laboratories, of which 7 are located at the Eastern Regional Research Laboratory, Wyndmoor, Pa., one is at Beltsville, Md., and two are at Washington, D. C. (of the Washington laboratories, one, the Dairy Products Laboratory, has part of its research program at Beltsville). Two of the Division's laboratories conduct pioneering research, one in animal proteins and the other research on allergens found in certain agricultural products.

In every phase of their research, Division scientists cooperate with representatives of colleges and universities, State Experiment Stations, research institutes and associations and industrial organizations. Much of the cooperation is informal, but some work is conducted under conditions described in written cooperative agreements and memorandums of understanding. Currently 7 such agreements are in effect.

The farm products with which the Eastern Division deals provide more than half of the nation's cash farm receipts. The major part of U. S. farmland suitable for cultivation is used to provide feed for livestock and dairy cattle; in seven states tobacco provides more cash receipts than any other field crop. Hence it is clear that maintaining and enlarging the markets for these farm products should be a major national concern.

The opportunities are great. A striking illustration is provided by meat and milk: a one percent increase in meat consumption, which might be achieved through improved quality, lower processing costs and new product development, would result in an increase in feed use equivalent to 80 million bushels of corn. Similarly, a one percent

increase in milk consumption would result in an increase in feed use equivalent to 25 million bushels of corn.

Division scientists have already achieved much, both in terms of discoveries now commercialized and discoveries of a fundamental nature that will be exploited in the future. Some commercialized developments based on the research of Division scientists are summarized here:

Potato flakes. Production of potato flakes, a form of dehydrated mashed potatoes, is now carried out in 13 factories located in the principal potato growing areas. The factories have a total capacity of 60 million pounds of product, equivalent to 7 million bushels of potatoes. This development is one of several credited with reversing the downward trend in per capita potato consumption, to the benefit of potato growers.

Glutaraldehyde-tanned leather. Research on the chemical modification of hides and skins has led to the use of glutaraldehyde in making leather from cattlehides and sheepskins. The leather is used in the manufacture of shoes and garments. It is flexible and attractive and has unusual resistance to the deteriorating action of perspiration and alkaline substances such as soaps. Present annual production of glutaraldehyde-tanned leather is estimated at 15 million square feet, and is increasing. The development illustrates the defense of a traditional market against the competition of synthetics made from non-agricultural raw materials, to the benefit of livestock producers.

Maple sirup. Improvements in the technology of processing maple sap to sirup have raised both the quality and yield of sirup. Sanitation of the sap collecting equipment is a key factor. Paraformaldehyde pellets inserted in the tree taphole prevent bacterial contamination and fermentation of the sap, and also prevent premature clogging of the taphole by microorganisms. In this way, sap quality is kept high and more of it is obtained. In a recent year 80 percent of the maple sirup produced was of the two top (light colored) grades, whereas previously not more than 50% was in those grades. Also, trees treated with the paraformaldehyde pellets yielded from 17% to 77% more sap than untreated trees. This development will materially increase returns to maple producers without additional investment in capital or labor.

Pasteurizer-deodorizer. A new low-cost process that pasteurizes and deodorizes market milk at temperatures no higher than those required for pasteurization, provides improvement over existing procedures for deodorizing milk. Market milk is brought to pasteurization temperatures

(161-165° F.) by steam injection, then deodorized by flash cooling in a vacuum chamber. This removes volatile feed flavors that may have been present in the milk, along with the water added as steam. Although some milk has been deodorized, the new process is more efficient and less costly than conventional processes. Some of the essential features of the new process have been adopted by a large dairy that handles 31,000 quarts of milk per hour.

Industrial chemicals from fats and oils. Basic and applied research in the chemistry of animal fats has led to the industrial use of many chemicals based on animal fats and vegetable oils. These uses include epoxidized oils as stabilizer-plasticizers in vinyl resins, vinyl stearate polymers and copolymers in waxes and water-emulsion paints, derivatives of α-sulfoacids in wetting agents and soap-detergent combinations, improved oleic acid as a raw material for the chemical industry and improved emulsifiers used in the manufacture of synthetic rubber.

The volume of fats and oils going into these markets is estimated at about 100 million pounds per year.

It is evident from these examples that the Eastern Division can make highly valuable contributions to agriculture. Indeed, it has been estimated that for utilization research as a whole--adding together the contributions of the Eastern, Northern, Southern and Western Utilization Research and Development Divisions--some $2.5 billion has been added to the value of products made as a result of the product or process developments of utilization research. This addition represents more than $12 worth of benefits for every dollar spent on utilization research and development in the Department.

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In this report, there is presented a description of the Division's program as of July 1, 1962, and a summary of progress for the period July 1, 1960 through June 30, 1962. The Research and Marketing Advisory Committees, in their meetings during fiscal 1962, devoted their attention to a review of the research program of the Department, hence this report provides information on progress to cover the period since the last report on progress to the Committees.

AREA NO. 1 AND AREA NO. 2. DAIRY PRODUCTS - CHEMICAL, PHYSICAL AND BACTERIOLOGICAL CHARACTERISTICS; DEVELOPMENT OF NEW AND IMPROVED PRODUCTS AND PROCESSING METHODS

Problem. Dairying is one of the largest segments of American agriculture: dairy products represent 13.9 percent of all farm cash receipts; milk production requires 140 billion feed units annually; milk is a highly nutritious food. It is clear from these facts that research which succeeds in increasing the consumption of milk will have far-ranging effects in raising nutritional levels, in increasing farmers' income, and in increasing consumption of feeds. There is opportunity to increase milk consumption, for per capita consumption is currently at its lowest point in over 30 years at 622 pounds, whole milk equivalent. Current consumption in the U. S. is well below that of several foreign nations, including New Zealand, Canada, Australia, Sweden, Norway and the United Kingdom.

Increased consumption can result from improved quality of manufactured dairy products, from cost reductions based on improved processing technology, from the development of new products, or from any combination of these. The development of new and improved processes and products is the objective of utilization research on dairy products.

Both basic and applied research in this field are needed; applied research is the direct antecedent to the development of new products and processes, and basic research provides the information which permits applied research to proceed most effectively.

Increased emphasis on basic research has been advocated by the Commission on Increased Industrial Use of Agricultural Products, and the National Agricultural Research Advisory Committee. Basic research is considered primarily the responsibility of public agencies which disseminate their findings for use by all.

One aspect of the problem posed by dairy products is the great need for fundamental information on the complex biophysical-chemical system which each dairy product is. The development of new products and new processing technology through applied research represents the exploitation of fundamental information. Such exploitation and development cannot continue indefinitely; the supply of fundamental information must be maintained and enlarged, and this is the purpose of basic research. The complexity of milk makes necessary the employment of several scientific disciplines in basic research on this commodity. These disciplines undertake investigations needed to identify and measure the amounts of individual chemical components present; the molecular structure of these components; how the molecules react; and the forces which determine the course of the reactions. These studies should be intensified. Other needed investigations include study of

the mechanism of the synthesis of milk; the properties of milk fat; and the factors responsible for the flavor of dairy products and the changes in flavor which occur during processing and storage.

There is also need for a vigorous and sustained program of applied research which is aimed to increase consumption of dairy products. Such a research program could stimulate consumption by development of products with increased palatability, convenience, or extended shelf life. Another opportunity is the possibility of developing new and improved processing technology which will reduce costs. Because the price elasticity of milk and milk products is greater than that of most food crops, cost reduction is an attractive avenue for increasing consumption.

Still another opportunity is the development of new milk products of low fat content, for example, a low-fat Cheddar cheese. Such a development could alleviate problems posed by current controversy over the effect of animal fats in the diet.

Increased consumption, however achieved, should have a powerful upward effect on feed consumption. It is estimated that milk production requires 140 billion feed units annually (a feed unit is the feed value equivalent to a pound of shelled corn). Hence a 1 percent increase in production would require feed equivalent to 25 million bushels of corn--the production of some 500,000 acres. If the feed were supplied by cropland pasture, more than a million acres would be needed.

It is thus manifest that utilization research leading to product and process development can provide a powerful stimulus to American agriculture.

USDA PROGRAM

The Department has a continuing long-term program involving chemists, biochemists, microbiologists, food technologists, and engineers, engaged in basic research on the composition and properties of milk, and in applied research directed to the development of new and improved dairy products and processing technology.

The Department's research facilities are located in Wyndmoor, Pennsylvania, Washington, D. C. and Beltsville, Maryland. The Federal scientific effort devoted to research in this area totals 82.6 professional man-years. The effort is distributed as follows:

(a) Work on composition and physical properties of milk involves 0.5 professional man-year at Wyndmoor, in a study of genetically-caused variation in the composition(and hence the interactions) of selected milk proteins. This work is cooperative with the Animal Husbandry Research Division, ARS. In addition, work sponsored by the Department

under a P.L. 480 grant goes forward at the Centre de Recherches sur les Macromolecules, Strasbourg, France (0.4 p.m.y.) on the structure of nucleic acids.

(b) Work on the isolation, structure and properties of milk proteins, and the interactions of milk components involves 19.5 p.m.y., at Wyndmoor, in pioneering studies of milk proteins and investigations of the reactions of proteins and polypeptides with each other and with other milk components. Cooperation is maintained with the Animal Husbandry Research Division, ARS.

In addition, research sponsored by the Department under P.L. 480 grants is under way at (1) National Institute of Agronomic Research, Paris, France (0.7 p.m.y.) on the activity of rennin toward individual components of casein.

(c) Work on flavor components and flavor stability of milk and milk products involves 7.5 p.m.y. at Washington. In addition, research sponsored by the Department under a P.L. 480 grant goes forward at the Biochemical Institute, Helsinki, Finland (2.5 p.m.y.), on the transmission of flavor components and other biologically-active compounds of feed of dairy cattle to the milk and milk products.

(d) Work on the microbiology of dairy fermentation involves 2.5 p.m.y. at Washington, in fundamental studies of the species, strains and biochemical activities of microorganisms used as starters in making butter, cheese, sour cream and cultured milks. In addition, research sponsored by the Department under a P.L. 480 grant goes forward at the National Institute for Research in Dairying, Reading, England, (0.7 p.m.y.) on differentiation of microorganisms important in dairy products.

(e) Research on the control of spoilage organisms involves 1.0 p.m.y. at Washington, in basic studies of the formation, germination and heat resistance of bacterial spores.

(f) Work on milk enzymes involves 1.0 p.m.y. at Wyndmoor. In addition, research on milk enzymes sponsored by the Department under P.L. 480 grants goes forward at the Instituto Nacional de Technologia, Rio de Janeiro, Brazil (0.6 p.m.y.), on proteolytic enzymes.

(g) Pioneering research on the allergens of milk and other agricultural products involves 4.0 p.m.y. at Washington.

(h) Research on dry whole milk and other dried milk products involves 30.5 p.m.y. at Washington and Wyndmoor. The program includes fundamental and applied research directed toward developing technically and economically feasible methods for producing dried whole milk with instant dispersibility and with flavor which will remain stable during six months storage at room temperature.

(i) Study of improved processing of fluid milk, cottage cheese and associated products involves 1.5 p.m.y. at Beltsville.

In addition, research sponsored by the Department under a P.L. 480 grant is going forward at the Centro Experimental del Frio, Madrid, Spain (0.4 p.m.y.) on protein destabilization in frozen milk.

(j) Research on improved ripened cheeses and cheese technology involves 0.5 p.m.y. at Beltsville. Contract research on improvements in cheese manufacturing technology is being conducted at the Ohio Agricultural Experiment Station, Wooster, Ohio; 0.3 p.m.y. is involved.

In addition, research sponsored by the Department under P.L. 480 grants is going forward at: (1) Institute of Biochemistry, Turku, Finland (0.6 p.m.y.) on growth-promoting factors for lactic acid bacteria in cheese making.(2) Kaira District Cooperative Milk Producers Union, Ltd., Anand, India (0.6 p.m.y.) on addition of non-fat dry milk solids to buffalo milk in the manufacture of hard cheese. (3) Institute of Dairy Industry, Warsaw, Poland (0.1 p.m.y.), on increasing the vitamin B content of cheese. (4) National Dairy Research Institute, Karnal, Punjab, India (0.5 p.m.y.) on milk coagulating enzymes of microbial origin, for cheese manufacture.

(k) Work on improved concentrated milks involves 2.0 p.m.y. at Washington. Contract research is under way at the University of Illinois, Urbana on possible flavor improvements in concentrated milk; 0.3 p.m.y. is involved. Contract research at Oregon State University, Corvallis deals with methodology for evaluating concentrated milks; 0.4 p.m.y. is involved.

In addition, research sponsored by the Department under a P.L. 480 grant is going forward at (1) Stazione Sperimentale del Freddo, Milan, Italy (0.9 p.m.y.), on new food products from concentrated milk and fruit juices suited to European tastes. (2) Institute National de la Recherche Agronomique, Paris, France,(1.0 p.m.y.) on non-protein nitrogeneous substances formed from milk proteins during processing. (3) National Institute for Research in Dairying, Reading, England, (0.7 p.m.y.) on the isolation and characterization of selected enzymes of milk to obtain fundamental information useful in improving the quality of dairy products.

(l) Work on improved butter involves 1.0 p.m.y. at Washington. Contract research at Iowa State College, Ames is concerned with the stability of butter and involves 0.2 p.m.y.

(m) Research on the identification and removal of radioactive nuclides from milk involves 9.5 p.m.y. at Beltsville. This is a cooperative program with the U.S. Public Health Service and Atomic Energy Commission, to develop effective ways for removing cationic radionuclides (Sr-90, Cs-137, and Ba-140).

RELATED PROGRAMS OF STATE EXPERIMENT STATIONS AND INDUSTRY

State Experiment Stations in 1961 reported a total of 67.6 professional man years distributed as follows: chemical composition and physical properties of milk and milk proteins, 10.4; chemical and physical properties of milk fat, 5.2; flavor components of dairy products and flavor stability, 8.5; microbiology of milk and dairy products, 13.2; allergens of milk, 0.3; enzymes of milk, 2.1; dry whole milks and other dried milk products, 5.7; improved processing of fluid and concentrated milk, cottage cheese and associated products, 7.0; improved ripened cheeses and cheese technology, 6.2; improvements in butter, 1.8; improvements in ice cream, frozen desserts and ice cream technology, 4.3; new and improved milk-containing food products, 1.4; utilization of whey, 0.8; removal of radionuclides from milk, 0.9.

Industry and other organizations also conduct research of interest and value on milk and dairy products. Industry is generally dependent on government, university, and other nonprofit institutional research for basic investigations into the chemical, physical and bacteriological characteristics of dairy products. Most of their effort is in development, with more attention given to technological research. Industrial research is being conducted to improve the dispersibility and flavor stability of spray dried whole milk. Industrial research in the fluid milk area is concerned largely with cost savings activities, container testing and low calorie formulation. A continuing program of cheese research is directed toward labor saving and attempts to shorten the ripening period. Industry has sought to develop an improved evaporated milk using the high temperature-short time sterilization process, and has supported work to evaluate concentrated milk in infant feeding. Industry has been active in developing new uses for milk in foods; bakery goods, cereals, confections and sterilized foods containing milk have been formulated. Estimated annual expenditures for industrial research in the dairy industry are equivalent to about 300 professional man-years distributed among at least 18 different company laboratories; estimated industrial expenditures for basic research on milk are equivalent to perhaps 5 professional man-years.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Composition and physical properties of milk.

Genetics and its relation to chemical composition of milk is a new field of research which has great potential possibilities in future improvements of dairy products. Preliminary investigations on milk from individual cows have demonstrated a variation in stability to the development of oxidized flavor at refrigeration temperatures. Variation in the processing behavior of milks from month to month and season to season may in part depend on genetic factors. Some definite

knowledge is now available to show that the amino acid structure of the two known β-lactoglobulins has a genetic cause. Derivatives of β-lactoglobulin have been prepared through chemical reactions which will expedite studies of the structure of this milk protein.

B. Isolation, structure and properties of milk proteins; interactions of milk components.

1. Chemical and physical properties. The importance of milk proteins in milk products is established. Whereas a few years ago these proteins were talked of only as the caseins and whey proteins, now their complexity is becoming fully recognized. Advances in protein research tools permit the realization that milk not only contains a great number of different proteins, but also that they are highly reactive. Heat treatments normal to milk processing produce detectable changes in many of them; some of these changes are responsible for undesirable behavior in dairy products, as gelation in concentrated milks.

The existence of two β-lactoglobulins was confirmed several years ago; the minor differences in amino acid composition are known, and amino acid sequence is partially established.

Recognition of kappa-casein as a milk protein is now several years old; its reported power to stabilize milk concentrates against physical change may prove to be very valuable in designing successful commercial milk concentrates, but more thorough studies must still be made. Recent improvements in procedures for its isolation and purification will make these studies possible.

The presence in milk of an iron-containing protein in equilibrium with an iron-free protein is now established. Significant progress has taken place in the elucidation of the primary structure of α-lactalbumin; through partial hydrolysis with the enzymes pepsin, trypsin and carboxypeptidase followed by careful analysis of the resulting peptide mixtures a significant portion of its amino acid structure is known. Additional data have accumulated on two minor proteins of the casein complex--β- and γ-caseins.

A neutral protein was isolated from the fat plasma interface region of milk and shown to be highly antigenic. The usual tests for homogeneity indicated that this protein is a single entity.

As the sensitivity of tests for homogeneity improve, purity of some proteins has become suspect. In some cases indications of multiple composition become more frequent; sometimes these are traced to the association or aggregation of smaller unit molecules into polymers.

2. Interaction of milk components. The major goal of utilization research on milk processing is to obtain concentrated or dry milk which will reconstitute to a product as much like fresh milk as

possible. Failure to achieve this goal completely can be attributed chiefly to physical and chemical reactions between the components of milk; some possible reactions are: one protein with another, protein with lactose or protein with fat. Basic research to establish the nature of these interactions is essential to development of successful products.

The casein of milk is a complex mixture of proteins containing phosphates, calcium and citrate; inorganic ions, hydrogen ion concentration and temperature are also intimately concerned with physical behavior of milk products. Attack on this problem has followed two approaches; (1) model systems in which the effect of varying one component at a time is studied; (2) attempts to detect changes in whole milk by refined analytical techniques.

One casein fraction is easily coagulated by an increase in calcium ions; kappa-casein will keep this phenomenon from happening. Investigation of this interaction has been undertaken in the belief that it is vital to the development of understanding of the mechanism of thickening and other manifestations of instability. Polyphosphates also prevent the coagulation of calcium sensitive casein as well as whole milk under sterilizing conditions and subsequent storage. The interactions involved in this behavior are poorly understood. After homogenization of milk at pressures in the 6,000 to 8,000 pound per square inch range, much of the fat centrifuges to the bottom rather than to the top layer; the sediment is a combination of fat and protein. Heat is known to produce reaction of sugar and protein to give colored products; these may also be related to undesirable flavor changes.

C. Flavor components of milk; flavor stability.

Research on the cause of oxidized flavor in dairy products continues not only in the Dairy Products Laboratory but in several other laboratories as well. Contract research with Iowa State University on oxidized flavor in butter established that the offending cause was a compound so potent in producing oxidized flavor that even with the superlative powers of gas chromatography its nature could only be qualitatively identified since it gave no peak on the chromatograph record with the quantities at hand, but issued from the chromatograph with the unsaturated aldehydes containing 7 or 8 carbon atoms. Australian workers have just announced the composition of this compound as vinyl n-amyl ketone, an 8-carbon unsaturated carbonyl-containing substance. This story illustrates the vagaries of flavor research.

Stale flavor is another objectional characteristic which develops in milk powder and evaporated milk after long storage, even in low oxygen concentration. The chemical nature of this flavor is even less well defined than that of oxidized flavor. Utilization research has active investigations in this area both in its own laboratories and by contract. Just as genetics is already known to control the structure of β-lacto-

globulin in milk, so may similar laws control one or more flavor genes in milk secretion. Therefore, for a better understanding of milk flavor the goal may be achieved more quickly with individual cows; but even here factors such as feed and lactation period also have an effect on total flavor. Development of analytical techniques for separation and identification of individual flavor components is progressing satisfactorily; however, the complexity of the problem is such that several years of coordinated effort may be needed to unravel it.

One aspect of research in fluid milk, sterile milks and reconstituted powders that has received little attention is the reaction of the consumer to the different flavors that occur. The industry has an established scoring system with trained tasters. By contract with Oregon State University data on consumer evaluation of milk flavors are being collected on the same samples that will be scored by a trained taste panel.

Preliminary results indicate good correlation between the two types of panel; that is, a sample with a flavor disliked by the consumer will get a low score on a trained panel.

D. Microbiology of dairy fermentation.

Cheddar and cottage cheeses are the major products in this category. The chief problem with these products is need for greater uniformity and for intensification of desirable flavor, a phenomenon resulting from microbial reactions; the chemical reactions giving off flavors so characteristic of milk concentrates and powders are not such serious problems in cheeses. Although several studies on the chemical composition of cheese flavor have appeared, duplication of natural flavor with synthetic mixtures has not been completely successful. Attempts to control the flavor of cheese have been trial and error type experiments. Progress in this area depends upon coordinated basic research--both chemical and microbial--to secure a better understanding of the process which accompanies flavor genesis.

Other developments in cheese technology concern the control of so-called starter failures. Two causes are involved: (1) the use of antibiotics in the control of disease, which may result in contamination of the milk with chemical substances to which certain cheese starter organisms are especially sensitive; (2) contamination of starter cultures with bacteriophage (phage), a disease of microbes. Investigation of this problem in commercial cheese factories has established that phage is the major cause of starter failure and that it can be prevented by the use of special phosphated media for the propagation of starter cultures.

E. Control of spoilage organisms.

For many years the dairy industry has recognized that bacterial spores make necessary the high temperatures needed to sterilize fluid milk

products. If means were available to make these resistant forms of bacteria more labile to heat, then fluid milk products could be sterilized without producing the strong heated flavor which is objectionable to many consumers. If this problem is to be solved, basic research on the nature of the spores' unusual heat resistance must precede their practical control with temperature treatments comparable to those for the vegetative forms of bacteria. One chemical difference between vegetative and sporulated forms of bacteria is the presence in the latter of significant proportions of a compound called dipicolinic acid, a compound not previously recognized as having a part in biological processes. Present research is studying how this substance is tied up with the unusual heat stability of spores.

F. Enzymes in milk.

Milk contains several enzymes; all are sensitive in varying degrees to heat. The lipolytic enzymes produce undesirable flavor changes in homogenized milk unless they are destroyed by a heat treatment. Alkaline phosphatase is the basis of the test for adequate pasteurization of milk.

The Milk Properties Laboratory is investigating the purification of an enzyme called acid phosphatase which, under suitable conditions, particularly of hydrogen ion concentration, hydrolyzes the organic phosphorous bonds of those caseins containing this element. Small amounts of the pure enzyme have been prepared from milk by chromatographic procedures. When more material is available its properties and reactions will be studied.

G. Allergens of milk and other agricultural products.

Many infant ailments are often diagnosed superficially as milk allergy. More sensitive tests for allergic reactions show that the majority of these reported reactions of milk have some other explanation; yet there is an occasional authentic allergic reaction to milk. The Allergens Pioneering Laboratory program concerns the characteristics of the allergens of cow's milk and, where possible, their chemical nature, and how they can be inactivated by commercial processing techniques. The rarity of human subjects who have a real sensitivity to milk allergens and the lack of other suitable methods of study has made progress in this field slow. Recent isolation of a milk protein with strong antigenic properties may expedite this program.

Progress on practical aspects of allergens in agricultural products depends to a considerable degree upon the amount of basic information on immunological processes which is available. A method was developed for the detection of dextran in animal tissues which is based on a phenomenon similar to reversed passive anaphylaxis. Treatment of castor bean or cottonseed allergens with certain chemicals or enzymes modifies their allergenic properties as well as their chemical

composition; however, most treatments leave some residual activity which may, of course, be from a small amount of unchanged original material. Heating castor beans with aqueous calcium hydroxide destroys their allergenic activity and also the toxicity of ricin, a toxic protein in this agricultural product.

H. Dry whole milk and other dried milk products.

Foam drying continues to look promising for preparing easily dispersible whole milk powder of beverage quality. Some technical problems remain, but there is confidence that they will be solved. Further improvement in flavor stability during storage is desirable.

1. Process development. Four methods for the continuous drying of milk foams have been evaluated. Two methods, the vacuum and the spray, warrant further extensive investigation.

A cost estimate for drying whole milk by the spray foam process has been prepared. A similar cost estimate for the continuous vacuum foam drying process has been brought up-to-date. Both estimates are provisional. The estimates suggest that milk dried by either process can be sold profitably in retail stores at a price considerably lower than the average store price for fluid milk. Since in several respects relating to quality there are now important differences between the spray and vacuum dried milks, it is intended to continue research on both drying methods until the evidence is complete enough to allow a sound decision between the two to be made.

Two other methods of drying whole milk have shown little promise on quality or economic grounds and research on them has been terminated. These are atmospheric drying of milk foams on a moving belt ("foam mat drying"), and spray drying in an inert atmosphere.

2. Spray drying. Spray drying is particularly attractive because it is inherently cheap and as conventionally practiced is widely familiar to the milk processing industry. Our own recent experiments designed to spray dry and agglomerate foamed concentrated milk in a single pass through the dryer have been so successful as to give rise to a feeling of considerable optimism concerning this mode of drying. Although the fluid foam is formed by the expansion of inert nitrogen gas incorporated at high pressure into the whole milk concentrate, the drying of the foam droplets is accomplished in a current of hot air. A fine cellular structure is characteristic of the dried particles. In practically all aspects of initial quality, spray dried foams seem to be just about as good as vacuum dried foams. With the existing spray dryer, powders with 3% residual moisture content have been produced at rates up to 400 pounds per hour. The output when drying foams is significantly higher than has been possible in conventional operation.

Although the process as practiced is carried out in air, no adverse

effect on flavor stability from exposure to hot air in the dryer has yet been recognized, and no chemical evidence for oxidative damage to the milk fat has been found.

Important problems to be solved include finding means for: quickly cooling and deoxygenating the dry powder, and packaging it under oxygen-free conditions required for satisfactory flavor storage life; reducing the tendency toward forming a persistent froth on reconstitution; and lessening the variability in keeping quality of powders with seemingly identical origin and history.

Lenthening storage life by controlling development of "stale flavor," and improvement of "self-dispersion" or "spontaneous solution" are problems common to both the spray and vacuum processes.

3. Continuous vacuum drying. Continuous vacuum drying on a moving solid belt has been elaborated from the original batch process for dehydrating whole milk concentrates in the form of a fine-grained foam. Dehydration is relatively rapid--the transit time being of the order of a minute--and is carried on in the virtual absence of oxygen, which minimizes oxidative damage during drying and should greatly facilitate inert gas packaging of the dry foam in a continuous commercial operation. Economic feasibility of this process now appears to require "boildown" of the milk concentrate foam in order to considerably increase the dryer output. The intent of the boildown is to remove most of the water in the milk concentrate through initial violent boiling, after which the residue collapses to a thin fine-grained foam in good thermal contact with the stainless steel belt. From such a foam it should be possible to remove the residual water at a rate which would improve the overall production of dry milk foam to a worth-while degree. Boildown also results in an increase in powder density to 0.35 g./cc or higher, with a resulting large saving in packaging expense. Experience extending over three years indicates that the type of foam instability which permits boildown is a seasonal characteristic, and is manifested to the most favorable degree during the winter months of December and January.

Since controlled foam stability is essential to the success of the boildown technique in the continuous dryer, a laboratory investigation of milk concentrate foams has been undertaken. A reproducible test has been developed for measuring extent of foaming and foam stability, and correlation of these properties with viscosity, temperature and surface tension has been studied. The effects of various additives are now being determined, as are the effects of the natural constituents which change to an important extent with season when these constituents are artificially varied in amount. Pending a general solution to the problem of controlling boildown, a statistically designed plan for determining the operating variables that will give maximum output from the continuous dryer is in abeyance.

The continuous vacuum process has the distinct advantage of permitting

virtually no exposure of the milk to oxygen from pasteurizer to package. At present, the continuous dryer is equipped only to deliver dried milk to polyethylene bags in a vacuum tight receiver. An enclosed packaging apparatus has now been built which will allow transferring the milk powder, by means of nitrogen-filled bags, from the receiver into a chamber filled with nitrogen, and comminuting and canning the powder in the inert atmosphere. The effect of this refinement on flavor stability will be tested. The apparatus will also be used in determining the practicality of several methods of in-package oxygen scavenging for reducing the level of residual oxygen still more.

4. Quality improvement. One of the essentials for preserving fresh flavor in foam-dried milks is storage in the almost complete absence of oxygen. A novel method for achieving and maintaining a "zero-oxygen" level over milk powder is being investigated extensively. The principle of the method is the in-package catalytic combination of oxygen with hydrogen by palladium at ordinary storage temperatures. The catalyst is a small pellet containing a few hundredths percent of palladium on a highly porous inert support. The cost appears to be economically feasible. In our experience reduction of headspace oxygen over canned spray dried milk foam from 1% to 0.003% by means of in-package hydrogen-palladium catalyst requires no more than a few hours at room temperature.

Development during storage of a stale flavor much like that of old evaporated milk continues to be our most annoying flavor problem in samples of whole milk powder packaged with an inert gas. This stale flavor is qualitatively distinguishable from the stale flavor sometimes found predominant in nonfat milk powders. "Whole milk powder stale flavor" presumably arises in great part from the milk fat, although the reactions that take place and the participation by proteins, milk sugar and other components are unknown. No means for preventing stale flavor development during storage is now apparent, except for use of impracticably low temperatures. Although exposure to water vapor promotes the stale flavor development, drying of milk powder to an abnormal extent has not yet resulted in a worth-while inhibition. An experiment in which whole milk powder is stored in the virtually complete absence of moisture will, it is hoped, show what potentiality exists for controlling stale flavor through the residual moisture content in the milk powder.

Increasing thought is being given to involvement of minor lipids, possibly originating in the rumen, which are precursors to off-flavor imparting substances by reactions in which oxygen does not participate.

In empirical attempts to improve flavor characteristics, variously purified and fractionated milk fats have been emulsified with skim milk concentrate and foam dried. Powders made with milk fat which had been deodorized or purified by steam or chemical treatment have not yet shown significantly improved storage stability. However, powders made

with the fraction of milk fat which is fluid at 70°F. have maintained superior flavor scores in a limited number of tests. This inquiry will be continued and broadened. Although it is possible in practice to prevent oxidized flavor from developing in dry milk by rigorous exclusion of oxygen during production and storage, it may be desirable to incorporate positive protection from autoxidation in order to extend the shelf life after the container of milk powder is opened to the air, as well as to reduce production and packaging costs. Hence our continuing interest in added and self-developed antioxidants. A measure of success in securing antioxidant activity has been achieved both by mildly heating all of the milk to be dried and by adding a small proportion of much more strongly heated whole or skim milk concentrate to the remainder of the milk to be dried. The effective antioxidant so produced is not known and its identification may be a worth-while research endeavor. Mercapto groups derived from serum proteins are often identified, very likely incorrectly, as the active antioxidant. Low molecular weight sulfur compounds probably contribute most to the moderately objectionable "cooked" flavor which develops at the same time but not necessarily at the same rate as the desired antioxidant property.

Because milk fat is implicated in the more important off-flavors, it is of interest to know to what extent the keeping quality of milk powder can be improved by reducing its fat content. Our observations show that over the fat content range from about 14% to 40%--the normal is 26%--there is no important difference in flavor stability. With a fat content in the powder of less than about 14% the reconstituted milk is, to the palates of most people, deficient in whole milk character to an unacceptable degree.

An attempt is being made to obtain through contract a meaningful expression of consumer reaction to types and intensities of off-flavors which develop most frequently in whole milk powders, in order to evaluate the scoring system which the trained taste panels use. The off-flavors of greatest concern are "stale," "oxidized," "cooked," and "astringent."

To assist in the investigation of factors which affect quality characteristics such as ease and completeness of milk powder dispersion, persistence of froth on reconstitution, and variability in the tendency to develop off-flavors on storage, a simple all-glass apparatus has been built in which vacuum dried foams can be made from as little as a pint of milk. A striking observation made possible by this apparatus is that some powders prepared from the milk of individual cows have very good flavor immediately on removal from the dryer, but show off-flavor to a serious degree when tasted after exposure to air for as little as three hours. Other individual milk powders retain completely their initial fine flavor.

5. Structure and dispersibility. Work on the protein factors which relate to the dispersibility of milk powders has continued. Attempts

are being made to characterize the configurational changes in molecules of the casein fraction during uptake of limited amounts of water which result in loss of milk powder solubility. Apparently a low molecular weight protein dissociates during this destabilization process. Additional evidence was obtained which shows that foaming milk brings about a partial fractionation of micelle constituents, with lower molecular weight material moving into the films of the foam. A study is being made of the effect of oxidation on the constituents of the casein micelle, and the role of phospholipids in the stabilization of the micelles during dehydration and rehydration is being investigated.

Gas adsorption studies were carried out to determine the specific surface area and capillarity of surfaces produced by various drying methods. The results when compared with results of permeametric measurements, revealed that only "instantized" skim milk powders possess surfaces characterized by capillary-like pores or crevices. The "free fat" globules of foam dried whole milk contribute appreciably to the surface area. Foam dried whole milk has a high specific surface area relative to foam dried skim milk. Fluorescence microscopy was used to determine the state of the fat in the interior of foam dried milk powder particles. Cross sections of plastic-embedded milk powders show the fat globules to be distributed more uniformly in foam dried milk than in conventional spray dried milk.

A method was developed, based on x-ray diffraction, for measuring the crystalline lactose content of milk powders.

A unique surface-active protein is present not only on the surface of the fat globules but also in the plasma of milk. The distribution of this protein in milk and the changes in distribution during the production and reconstitution of dry whole milk are under study. The protein contains a carbohydrate moiety which includes a high level of neuraminic acid, a compound of great interest in biochemistry. A non-dialyzable fragment has been isolated which contains almost equal amounts of carbohydrate and peptide. The fragment is being analyzed for its amino acid composition and immunochemical similarities to the glycomacropeptide of casein.

I. Improved processing of fluid milk.

Since fluid milk consumption represents about one-half of total milk production, any improvement in quality can easily lead to a significant increase in consumption. The introduction a few years ago of vacuum deodorization treatment to remove volatile feed or weed flavors gave a better fluid milk; however, pasteurization was performed in a separate step. The Dairy Products Laboratory has designed and successfully demonstrated equipment which pasteurizes and deodorizes milk simultaneously. This product has superior storage life in the refrigerator as compared with pasteurized raw milk. At least one manufacturer of dairy plant equipment has this design of equipment available

for installation in commercial dairy plants; some units have already been installed.

The decline in milk consumption and reasons proposed to account for this emphasize the need to make available to the consumer fluid milk of different composition than that of whole or nonfat milk. Technical knowledge to produce such products has been tested successfully; commercial interest should be stimulated.

J. Improved ripened cheeses and cheese technology.

Emphasis in cheese research has continued to stress basic chemical and bacteriological studies on the nature and development of the desirable flavor in Cheddar cheese, including composition and propagation of conventional and supplemental starters, lipolytic enzymes and means for controlling bacteriophage. Commercial enzymes--proteinases and lipases--were added individually in cheesemaking. No improvement in flavor or texture was found with proteinases. Rancid and unclean flavors developed with some lipases, such as steapsin. The addition of mildly lipolytic supplemental cultures to cheese milk and the controlled development of rancidity in the milk intended for cheese starters are the only means that have continued to show promise of increasing cheese flavor.

The phosphated nonfat dry milk is an effective control for bacteriophage in cheese manufacture and should result in significant savings for the industry. Cheese whey, especially that from cottage cheese, has been a problem to the industry for many years. Economical utilization has succeeded only for a small portion of the available products; disposal has entailed additional costs. A feasible process for production of yeast from whey was developed earlier. Recent animal feeding experiments by Pharmacology Laboratory, WU, have shown that this yeast from whey is equivalent to other food yeasts. This should open up profitable outlets for an additional portion of the available whey. Better utilization of whey requires its conversion to a dry product in order to minimize transportation costs. With cottage cheese whey this has not been possible due to its high acid content. A new process for drying whole milk also converts whey to a very attractive free flowing powder.

There is a definite need for low fat dairy products to satisfy those consumers who are diet conscious or who may have to avoid fats for some other reason. A skim milk cheese with the flavor characteristics of Cheddar cheese should find a ready market. Two problems remain unsolved in achieving such a product; the body and texture of experimental preparations made to date are not acceptable; nor has an attractive flavor of good Cheddar cheese been obtained as yet.

In several investigations dairy chemists have studied the composition of Cheddar cheese flavor. Other research indicates that the good

flavor of Cheddar cheese arises from the fat. Only by further research can it be established whether a low fat cheese can be developed with the customary desirable flavor.

In the manufacture of cheese, milk is usually coagulated with the enzyme rennet. To clarify its action on α-casein methods are under study for hydrolyzing quantitatively the carbohydrates from the glycomacropeptide resulting from the enzyme action. Heating for one hour in dilute sulfuric acid (pH 2), the usual procedure, did not hydrolyze the carbohydrates quantitatively.

K. Improved concentrated milks.

The previously announced discovery that some polyphosphates inhibit gelation in concentrated milks is believed to represent major progress toward solution of the physical instability problem which has been a serious hindrance to successful commercialization of high temperature-short time sterilized milk concentrates. With further experimentation since the initial discovery, facts are coming to light which show that the polyphosphates do not always stabilize milks against coagulation during the sterilizing treatment; partial hydrolysis of the polyphosphates to orthophosphate is a factor in this phenomenon. In non-sterile refrigerated and frozen milk concentrates stability to heat is not an important factor. The evaporated milk industry is actively studying the practical use of polyphosphates. Elucidation of the mechanism of the polyphosphate effect is under study in EU.

The importance of the fat-water interface to the physical stability of milk concentrates has been recognized for a long time. Research on this indistinctly defined area of milk has posed difficult experimental problems. A basic study of the effect of individual milk proteins on the energy relationships at this interface, as measured by surface tension, is contributing to knowledge from which better milk concentrates can be designed. The mucoprotein recently isolated from the fat globule membrane of milk also has a stabilizing influence in concentrated milk; other milk proteins, especially kappa-casein, likewise influence the physical stability of milk products and are still receiving research attention.

Flavor deterioration in concentrated milks usually results in a defect characterized as stale. Through a contract with the University of Illinois the chemical nature of this stale flavor is being investigated. Progress in this field is not as advanced as that on oxidized flavor. A more efficient procedure for separating the flavor from the product is badly needed before more rapid progress can be made in its identification.

L. Improved butter and milk fat products.

At the present price of butterfat there is some agreement among industry

and government representatives that butter will become an increasingly serious surplus problem unless some way is found to raise its consumption as human food. Foreseeable means to achieve this goal by research, even if successful, may hardly be sufficient to keep up with increases in the annual surplus. In cooperative research with EU the University of Maryland has investigated the best source of fat for ice cream. The product called concentrated sweetened cream, which was developed in DPL as a substitute fat source for frozen and other forms of cream, has been carefully studied. Results indicate that concentrated sweetened cream is at least equal to and possibly superior to other sources of milk fat in this major product.

M. Removal of radionuclides from milk.

Research on the removal of fallout from food, especially milk, continues to be a popular subject with public and political overtones. The utilization research program on the removal of strontium-90 from milk is a joint undertaking of the Atomic Energy Commission, Public Health Service and Agricultural Research Service. A successful procedure has been developed. Milk is acidified to convert all the strontium to a form which will be adsorbed on a cationic exchange resin; the added acid is finally neutralized to return the treated milk to its original pH. A portion of the dairy industry is already acquainted with the ion exchange process through manufacture of low sodium milk on a limited scale. The acidification and neutralization steps are not desirable; further intensive research is now in progress to achieve the same result without them. Cost of the process is being further reduced by additional refinements in techniques. Other methods have been proposed for the removal of strontium-90 from milk. In one of these milk is treated with calcium phosphate which is later removed by filtration or centrifugation; however, drastic changes in milk composition accompany the removal under all conditions so far fried. In addition, the necessity to use one pound of calcium phosphate for each pound of milk solids to achieve effective removal indicates that the seeming simplicity of the process does not necessarily make it less expensive than ion exchange. The Chemical Engineering Division of the Polytechnic Institute of Brooklyn has demonstrated in the Beltsville laboratories that strontium-90 can be effectively removed with an electrodialytic technique. The Civil Defense Division of the Department of Defense has given out a contract to a commercial concern to further develop this process which is based on a plastic membrane permeable to metal ions.

The Public Health Service has initiated nutritional and sanitary studies to gather information on any effects that the ion exchange removal process may have on milk. Two further developments are planned for fiscal 1963 by the Atomic Energy Commission, Public Health Service and Agricultural Research Service in the pilot plant at Beltsville. Glass columns will be supplemented with stainless steel ion exchange columns so that tests can be run under pressure;

equipment for automation of the process will be installed. Funds have been requested for installation of a 100,000 pound per day plant in industrial surroundings.

One of the important questions from the public and the dairy industry is how soon can milk plants install the equipment. A vital factor in this decision is beyond the responsibility of Utilization Research; the government agencies responsible for the safety of food must first decide when treatment is necessary; there is no indication that removal of strontium-90 will be required in the foreseeable future as a result of testing atomic weapons.

N. New drying technique for liquid foods.

A new technique for drying foods was developed by injecting an inert gas under pressure into concentrated cheese whey between the pump and the spray nozzle on a conventional milk dryer. Outstanding success with this product suggested possible extension to other dairy products and foods. A very promising field is dry whole milk; a product with excellent properties when freshly prepared is obtained. There is an increase in dryer capacity of 20 to 30%.

At least one manufacturer of nonfat dry milk has made commercial application of this foam spray drying principle, producing as much as 20,000 pounds of powder per day; he is installing additional equipment. Planned adoption of this technique for nonfat milk and other products is reported, including cultured sour cream and blue cheese in the dairy field. Interest is evident for possible application to eggs and citrus juices.

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AREA No. 3 - MEAT - PROCESSING AND PRODUCTS

Problem. Livestock production is the greatest single source of farm income: production of meat animals accounted for 31 percent of farm cash receipts in 1960, and the major part of U. S. farm land suitable for cultivation is used to produce feed for livestock. Hence any research which succeeds in stimulating an increase in the consumption of meat and other livestock products can provide a powerful stimulus to U. S. agriculture. For example, a one percent increase in meat consumption would require feed equivalent to about 80 million bushels of corn.

Increases in livestock consumption may be achieved through development of new or improved meat products, or through improved meat processing technology which results in lower costs. In addition, increases in the value of hides, animal fats and renderers' proteins will benefit the livestock industry by providing additional revenues which could permit reduction in meat prices (thus stimulating consumption) or which could flow back through the marketing channels in whole or in part to livestock growers and feed producers. For example, it is estimated that loss of the market for hides would cause an increase of meat prices that would result in a decrease of 2 percent in meat consumption. Such a decrease would eliminate a market for feed equivalent to 160 million bushels of corn. Conversely, an increase in hide values would operate in the opposite direction and would result in greater income to the livestock industry and in increased utilization of feed grains.

The attainment of an increase in livestock consumption requires both applied and basic research. Applied research is the forerunner of commercial practice and is an indispensible element in successful development. But applied research is based on the fundamental knowledge that is acquired through basic research, and represents the exploitation of this fundamental knowledge. The supply of fundamental knowledge must be maintained and expanded, if applied research is to be most effective and fruitful. The need for basic research has been pointed out by the Commission on Increased Industrial Use of Agricultural Products, by the National Agricultural Research Advisory Committee and by other responsible groups.

For the reasons outlined above, research which succeeds in increasing meat consumption can have a powerful effect on American agriculture. The potential effect may be assessed from the facts that (1) meat has a high elasticity of demand (a 1 percent drop in retail meat prices will result in a 0.7 percent increase in

consumption); (2) the production of 1 pound of livestock requires the equivalent of 7-8 pounds of feed grains; and (3) the U. S. per capita consumption (160 pounds) is no higher than it was 50 years ago and is below that of several other countries, including Australia (234 pounds per capita), New Zealand (222 pounds), Uruguay (234 pounds) and Argentina (166 pounds).

Increases in meat consumption may be achieved by the development of new and improved products that will stimulate demand, or by improvements in processing technology that will lead to reduction in meat prices. The effect of lower prices on meat consumption and on feed utilization is especially striking. Thus, a 1-cent-per-pound reduction in retail meat prices should lead to an increase in consumption of about 1.1 percent. The additional production required to meet this increase will require feed equivalent to about 85 million bushels of corn--the output of about 1.6 million acres.

The attainment of increased consumption will require a vigorous and balanced program of applied and basic research. There is a need for more applied research on processing and preservation, including time-temperature studies of frozen meat, and on new and improved meat products. Of equal or greater importance is the need for more basic research on the physical, chemical and organoleptic characteristics of meat, and the microbiology of meat.

USDA PROGRAM

The Department has a continuing program involving chemists, biochemists, and microbiologists engaged in both basic studies and the application of known principles to the solution of problems in the processing of meat and its products. The Department's research facilities are located at Beltsville, Maryland.

The Federal scientific effort devoted to research in this area totals 15.8 professional man years. This effort is applied as follows:

(a) Chemical and physical properties of meat proteins involves 3.0 p.m.y.

(b) Chemistry of meat fats and fat-protein interactions involves 3.5 p.m.y. at Beltsville. A research contract at Florida State University, Tallahassee, involves 0.3 p.m.y. in a study of the interactions of meat pigments with tissue lipids with the objective of developing methods to control rancidity and pigment fading.

(c) Study of improved meat products and processing methods involves 2.5 p.m.y. at Beltsville; a research contract at Michigan State University, East Lansing, involves 0.2 p.m.y. in a study of the

utilization of dry milk solids in sausages. In addition, research sponsored under P. L. 480 grants is under way at:

(1) Ministry of Agriculture, Cambridge, England (1.3 p.m.y.), in study of the accelerated freeze-drying of meat.

(2) Experiment Station for Food-Preserving Industries, Parma, Italy (0.8 p.m.y.) in study of the corning of beef.

(3) British Food Manufacturing Industries Research Association Surrey, England (0.7 p.m.y.), in study of pigment formation and color fixation during the curing of pork.

(d) Work on the chemistry of flavor of meat and meat products involves 2.5 p.m.y. at Beltsville.

(e) Research on the microbiology of meat and meat products involves 3.5 p.m.y. at Beltsville.

In addition, research sponsored under a P. L. 480 grant is under way at the Research Institute of Meat Technology, Finland (0.5 p.m.y.) in study of the influence of microorganisms on flavor development in sausage.

(f) Federal research on frozen meats and time-temperature tolerance is presently confined to a research contract involving 0.3 p.m.y. at Oklahoma State University, Stillwater, on tenderness reversion in frozen beef.

In addition, research sponsored under a P. L. 480 grant is under way at the Low Temperature Research Station, Cambridge, England (1.0 p.m.y.) on enzymes which attack connective animal tissues, for better understanding of freezer damage in frozen meat.

RELATED PROGRAMS OF STATE EXPERIMENT STATIONS AND INDUSTRY

State Experiment Stations in 1961 reported a total of 25.4 professional man years devoted to meat research and divided as follows: (a) Chemistry and physical properties of meat proteins, 6.8; (b) chemistry of meat fats and fat-protein interactions, 1.9; (c) improved meat products and processing methods, 8.3; (d) Chemistry of flavor of meat and meat products, 3.5; (e) microbiology of meat and meat products, 1.9; (f) Frozen meats: time, temperature, tolerance, 3.0. Industry and other organizations are also conducting valuable research programs on meat and meat products.

Most of the research effort of the American meat packing industry is directed to industrial utilization of by-products, but some research is conducted on better quality control devices; machinery

and automation for packaging, sausage making, variety and luncheon meat handling, and canned meats; improved formulations for canned comminuted meats; development of new products; use of tenderizing agents; studies on new curing and smoking methods; improvements in plant layout; and development of new slaughtering equipment and techniques. This program differs from the USDA program in that there is strong emphasis placed on development, and the solving of immediate problems, and comparatively little on basic research. Estimated annual expenditures for research on meat are equivalent to about 100 professional man-years. About 80 per cent of this effort is expended by about ten or a dozen of the larger firms.

There are hundreds of independent laboratories in the United States, and many work, at times, on small, short-term problems for clients in the meat industry. These problems can generally be classed as "fire fighting jobs". A few larger independent laboratories are more or less continuously engaged in some special development problems for the industry. Their work includes problems on sausage formulation, meat product additives, meat microbiology, by-product utilization, meat flavor studies, and package and machinery development. Some of the large foundations have similar interests, including one that works entirely in the meat field. The foundations have programs relating to nutritive values of meat, meat hygiene, meat product development, meat composition, and some fundamental studies. Estimated annual expenditures are equivalent to about 30 professional man-years.

Large food processing companies which are not primarily meat packers carry on some research on new product development such as dehydrated and/or frozen meat products, combination meat dishes, and meat canning. Estimated annual expenditures are equivalent to about 10 professional man-years.

The pharmaceutical industry carries on research in this field from two standpoints. One deals with the extraction of biologically active substances from meat by-products, such as hormones from glands. The other deals with the development of agents, such as antibiotics, for use in meat processing. By and large, the latter effort has been unsuccessful but remains active. Estimated annual expenditures are equivalent to about 20 professional man-years.

Chemical companies and equipment manufacturers are engaged in the development of new processing equipment, automation of processing, new packaging materials, development of chemicals as additives, new sanitizing agents for plant use, and development of special chemical by-products. Estimated annual expenditures are equivalent to about 10 professional man-years.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical and Physical Properties of Meat Proteins

A major protein in meat is myosin; its properties and composition can be readily changed by a number of factors. Two types of conformational changes have been detected; in the presence of lithium bromide or potassium iodoacetate the diffusion constant increased while the sedimentation rate decreased; although the myosin particles became less elongated, the molecular weight did not change. During storage myosin undergoes two simultaneous molecular changes: a portion disaggregates into a smaller molecule while other portions aggregate into two larger particles, probably a dimer and trimer.

Factors affecting the extraction of meat protein components have been investigated thoroughly. In sample preparation, grinding the meat was sufficient for general extraction. In the centrifugation step packing of the sediment gave an increase in total yield of protein as the rate of centrifugation increased. At least 85% of the soluble protein could be obtained with one extraction. Yield of extractable proteins depended upon salt concentration and hydrogen ion concentration. Over a wide span of pH and salt concentration swelling of ground meat was directly related to its content of salt-soluble and salt-insoluble proteins, but not that of the water-soluble proteins.

The protein extraction studies described above have been carried out using seven different muscles from the same carcass. The trend with pH was similar in all cases, but there were large differences in the values obtained from the different muscles. Variations in these properties were also measured on samples taken from one muscle at five intervals during aging for twenty-eight days. Marked changes were found in both magnitude and pH effect on the extraction of non-protein nitrogen and protein, and on the water retention of the residues.

To obtain basic information about the relation between tenderization of meat and proteolysis of the meat proteins by tenderizing enzymes a new physico-chemical method of analysis was developed. This method depends upon changes in the structure of meat when proteolytic enzymes split a few peptide bonds in the meat protein. In general, above pH 7 the initial rate of proteolysis increases in inverse proportion to the concentration of hydrogen ions. Also, 0.6 molar potassium chloride increased the reaction rate about 50%. Comparative studies with trypsin, chymotrypsin, papain, and a commercial tenderizer product were also made. Similar measurements, using myosin instead of beef muscle, gave results of a similar nature. Proteolytic enzymes find wide use in tenderizing meat and meat products.

Current methods measuring tenderization involve panel studies, mechanical devices or histological techniques that require large samples, or long time periods, or both. Only the histological techniques give any information about how the meat structure is modified by tenderization.

The moisture retaining characteristics of protein in meat processing, and an understanding of the variation of these characteristics with respect to ionic strength, pH, and inherent differences among the several fibrillar proteins, are essential to improving existing processing techniques and devising new ones. Better methodology for studying water holding is considered of primary importance in the industry to the improvement of many process control methods.

B. Chemistry of meat fats; fat-protein interactions

Organic compounds containing the carbonyl group are responsible for many of the undesirable flavors in stored meats and meat products. Presumably these objectionable substances arise from fat through a series of reactions which require oxygen at least as an initiator. Although this reaction can be delayed in some cases by the use of antioxidants, development of adequate protective measures awaits a better knowledge of all the products formed in these oxidation reactions and of the mechanisms by which they are formed. To date research on this project has developed improved methods of analysis for the complex mixtures of carbonyl compounds which result from the oxidation of fat. The more potent flavor producing compounds are relatively unstable and reaction conditions which give quantitative yields of derivative with one type of carbonyl compound may give low yields with another type, or even transform some into something else with entirely different flavor characteristics. These studies with such a mixture of compounds, present in meat or meat fat in only trace amounts, sometimes parts per billion are time consuming; but real progress is being made.

To speed up the fat oxidation process in the laboratory an inert powdered material, as celite or silica gel, has been coated with films of fat to which various potential promoters or inhibitors were added. Preliminary results with salt, lipoxidase and tocopherols showed differences in the pattern of carbonyl compounds formed. This project is developing fundamental information which has tremendous value in clarifying the mechanisms of oxidation or rancidification. Lipid oxidation deteriorates product quality, or at the very least, limits storage of many meat items, particularly such items as frozen meat, precooked meat dishes, and cured meat products, and thus is a problem of world wide importance and interest. Determination of the types of reactions involved affords a basis for developing protection against oxidative deterioration.

By contract with Florida State University a connection of meat pigments with lipid oxidation has been established. Cooking partially destroys the pigments and at the same time produces substances which tend to prevent lipid oxidation. Preliminary results indicate that both the antioxidant BHA and a tripolyphosphate ascorbate mixture protect cured meats from oxidation during freezer storage.

C. Improved meat products and processing methods

Addition of 3.5% nonfat dry milk to comminuted meat products increased yield and tensile strength, and had no effect on flavor or color; 10% added milk solids also gave an acceptable product. Use of fermented milk solids (dry whey) introduced a tangy flavor; nonfat dry milk also had to be added to achieve the needed water binding capacity. Adoption of these surplus foods as ingredients in sausage-like products could result in a significant increased use for them.

Increased fundamental knowledge of the chemical reactions involved in meat curing is essential to improvements in this type of product. Since the binding of electrolytes and water, and emulsification of fat, by meat proteins are recognized as essential reactions in acceptable comminuted meat products, studies were initiated to determine the effect of hydrogen ion and salt concentration upon this binding power. Centrifugation at 144,000 times gravity for 24 hours precipitated 99.5% of the soluble proteins. Binding capacity of salt soluble proteins was not affected by change in salt concentration at pH values over 6. Use of a technique devised for determining the emulsification capacity of tissue containing different amounts of "added water" showed that reduction in "added water" reduced the capacity of tissue to emulsify fat. Centrifugal fractionation of muscle suspensions in sucrose solutions and subsequent analysis showed the distribution of zinc in five fractions to be: filtrate, 1.23; structural tissue, 3.47; nuclei, 0.90; nitochondria, 0.06; and microsomes 0.05 milligrams zinc per 100 grams of sample.

The biochemistry of pork muscle pigments is the subject of a P. L. 480 grant to the British Food Manufacturing Industries Research Association. The meat pigment in oxidized form has been isolated in 70% purity. It forms a new pigment complex with nitric oxide, a gas which can be formed from reaction of the nitrite in curing agents with a reducing system in pork flesh. This new substance resembles the normal cured meat pigment, but differs in behavior, decomposing at once in contact with air. The rapid fading sometimes observed in hams after cutting might be associated with the presence of this pigment.

D. Chemistry of flavor of meat and meat products

Changes in the natural flavor of meat can be influenced by chemical reactions which occur normally during processing and storage, or by the use of properly selected microorganisms which result in formation or intensification of desirable flavor characteristics. Cured meats involve the addition of one or more salts which have an effect on growth of microorganisms; obviously if changes in flavor of cured meats are to be produced with microorganisms, strains must be selected which grow in the presence of varying concentrations of salts. Studies have been made with a large number of salt tolerant bacteria at salt concentrations varying from 0.5% to 12% sodium chloride.

The successful development of an inoculum for ham brines could have a profound effect on the acceptability of a major agricultural product which has been slowly losing its preferred status in the American diet. Better knowledge of the physiology of halophilic microorganisms is essential to such an achievement. In studies with 69 cultures differences in morphology at the two salt concentrations were recorded. Enzymes inhibited or not produced in media containing 12% sodium chloride were lipase and gelatinase and some of the enzymes involved in cleavage of sugars.

The important role of fats in meat flavor is recognized. Evidence collected in this research shows that phospholipids, one of the components of the fat fraction, do not contribute to desirable meat flavor, but that they may contribute to poor flavor.

One reason for restricted utilization of lamb and mutton is an objection that many consumers express for lamb flavor. A distinctly unpleasant volatile fraction has been isolated from lamb fat; elucidation of its chemical nature may lead to prevention of its formation or modification of its effect. In lamb flavor studies the lean portion of the meat has the same basic meaty aroma found to be characteristic of pork and beef. A fraction was isolated from lamb fat with a potent "muttony" flavor that was almost carbonyl free; thus, it is believed that both fat and water soluble components of the meat contribute to this flavor.

In these studies on flavor composition of meat extracts and volatiles important improvements have been made in analytical procedures, particularly in gas chromatographic techniques.

E. Microbiology of meat and meat products

Investigations on microbial activity at low temperatures involve a twofold approach. One is a study of the growth of the microorganisms. One of the enzyme systems that is active at temperatures considerably below that at which the microorganism will grow is the lipase, or fat-splitting enzyme.

By the use of known, mixed triglycerides the high degree of specificity of lipases from psychrophilic bacteria for the fatty acids in the 1-position has been confirmed. These enzymes were also found to be active at 0°F., the minimum temperature at which frozen foods are usually stored. Some activity was still evident within three weeks at -30°F. Analysis by gas chromatography of the fatty acids released showed a definite shift toward the unsaturated fatty acids as the temperature decreased.

Studies have also been made of the activity of lipases from other bacteria, yeast and molds. These microorganisms have been shown to vary considerably in the type of fatty acids split off from natural fats. Certain ones appear to have a greater affinity for palmitic and stearic acids while others more readily attack the unsaturated acids. The lipases from all microorganisms examined were active in frozen substrates even though some of the organisms would not grow below 50°F.

In order to study the nutritional requirements for lipase formation, a synthetic medium has been developed in which members of the genus Pseudomonas will grow and elaborate enzymes. Studies with thick-cell suspensions have permitted the investigation of an extracellular enzyme without the added complication of cell multiplication. This differentiation has indicated that the enzyme-synthesizing mechanism ("template") is present in the cell regardless of the conditions of cell growth. The functioning of this mechanism, however, is dependent upon the nutritional and physical conditions of the environment in which the cells are held. Thus, a food product might be held under conditions favoring bacterial growth but not lipase production and under subsequent conditions the lipase could be elaborated by preformed cells.

F. Frozen meats: time temperature tolerance

Studies conducted at Beltsville some years ago indicated that freezing had a marked tenderizing effect on beef steaks when they were frozen rapidly at low temperatures (-50° to -70°F.), thawed, cooked, and tested. When the steaks were stored in the freezer, as would be the case in practice, the initial gain in tenderness was soon lost. Further confirmation and explanation of this pehnomenon, and the development of means to prevent its occurrence, are the subject of a contract with Oklahoma State University.

Statistical analysis of taste panel ratings for tenderness and shear force values revealed no significant difference in tenderness between the unfrozen control steaks and those frozen and stored for the various times and temperatures indicated. Nevertheless, shear values were altered by the treatment stimuli. Steaks frozen at +15°F. showed an increase in shear values over the unfrozen control

steaks. This decline in tenderness became more pronounced as storage time was extended. Freezing at temperatures of 0°F. and below, however, produced a decrease in shear values of the experimental steaks. The data suggested that a part of this increased tenderness was forfeited during freezer storage; yet the samples frozen and stored for 12 weeks still sheared lower than their unfrozen controls. Decreasing freezer temperatures below 0°F. produced no consistent increase in tenderness.

Panel scores for flavor and overall rating indicated a preference for the frozen and stored product, whereas the juiciness values showed a significant decrease with storage time.

Through a P. L. 480 grant to the Low Temperature Research Station in Cambridge, England, the nature of connective tissue structure is being investigated, particularly the structure of the chondroitin sulphate protein complex. This complex consists of a central protein core around which are arranged many chondroitin sulphate chains in a structure resembling a spiral brush. The protein core carries a second polysaccharide, containing galactose and glucosamine, and it remains to discover how this polysaccharide is attached and the nature of the links between the chondroitin sulphate chains and the protein core. As would be expected by its structure, both hyaluronidase and several proteolytic enzymes attack the complex, but they degrade it in different ways. In the work on elastin it has been discovered that in some tissues the fibrous protein is intimately associated with a second system of proteins which contain sialic acid, hexosamine and neutral sugars and which appear to be combined with lipid in the native tissue. Some progress has been made toward isolating and separating this new family of mucoproteins, and it is believed that they are the substrates for the enzyme elastomucase which has a strong synergistic effect on the action of pancreatic elastase.

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* Work done under a P. L. 480 foreign research grant.

AREA NO. 4 - ANIMAL FATS AND OILS--INDUSTRIAL UTILIZATION

Problem. The 3.5 billion-pound-per-year output of inedible fats is one of the major products of the livestock industry. It also is one of major concern, because while production of animal fats has doubled in the last 15 years, its principal outlet (in soap) has declined sharply, and is still declining.

The best answer to the question of what to do with huge amount of fats is to find new uses through utilization research. Already utilization research has played a leading role in finding new uses for some 600 million pounds of animal fats, and thus helped retain markets for fats. There is need, however, for new uses not merely to retain or defend markets, but to expand them, and to upgrade the value of animal fats. The organic chemical industry presents a good opportunity for expanded markets, producing as it does a multitude of products--polymers, plasticizers, insecticides, herbicides, lubricants, paper chemicals--totaling 10 billion pounds. Animal fats possess "built-in" properties which make them potentially useful as raw materials to the chemical industry, but research must be done to realize this potential.

Both basic and applied research are needed; the basic to establish the fundamental facts as to composition, separation of constituents, and their chemical activity; the applied to put these facts to work in developing new industrial products.

An increase of 1 cent per pound in the value of animal fats would provide an additional revenue of $35 million to the livestock industry. This revenue will help the industry and growers in the same way as revenue from other animal products and by-products.

The attainment of an increase in livestock consumption requires both applied and basic research. Applied research is the forerunner of commercial practice and is an indispensable element in successful development. But applied research is based on the foundation of fundamental knowledge that is acquired through basic research, and represents the exploitation of this fundamental knowledge. The supply of fundamental knowledge must be maintained and expanded if applied research is to be most effective and fruitful. The need for basic research has been pointed out by the Commission on Increased Industrial Use of Agricultural Products, by the National Agricultural Research Advisory Committee and by other responsible groups.

USDA PROGRAM

The Department has a broad program of basic and applied research at Wyndmoor, Pennsylvania; and at additional locations where contract

research is being carried out involving chemistry and physics, aimed at developing new and improved products from fats for use in industry. Total man-years are 59.5. Of this, 24.0 p.m.y. are devoted to studies on chemical composition and the physical and chemical properties of animal fats at Wyndmoor. Studies involve fatty acid composition of animal fats using the latest techniques in chromatography and other techniques; intra- and intermolecular structure of pure components and derivatives and factors that influence development of off-flavors in fatty foods. A contract at Villanova University, Villanova, Pa., involving 0.3 p.m.y. deals with structural characteristics of purified triglycerides. Contract research on chemical and physical characteristics of organic peroxides involving 0.8 p.m.y. is going forward at the University of Pittsburgh, Pittsburgh, Pa.

Research on improved polymers, plastics, resins and lubricants involving 15.0 p.m.y. at Wyndmoor is conducted in the preparation of new products from fats through vinyl polymerization, condensation polymerization and synthesis of organic compounds for use as plastics, plasticizers and lubricants. Work on preparation of copolymers of octadecyl acrylate was completed during the report period. 5.0 p.m.y. are being devoted at Wyndmoor to research on development of improved synthetic detergents based on animal fats, which includes preparation of α-sulfofatty acids for use in synthetic detergents, and study of soap-detergent combinations for use as bar detergents. 14.0 p.m.y. are being devoted at Wyndmoor to exploratory investigations of new chemical derivatives of animal fats for use as chemical intermediates for industry. A contract with the University of Pennsylvania, Philadelphia, Pa., involving 0.4 p.m.y. provides for determining dielectric properties of long-chain fatty acid compounds and heats of combustion of certain fat compounds.

RELATED PROGRAMS OF STATE EXPERIMENT STATIONS AND INDUSTRY

State Experiment Stations in 1961 reported they were devoting 1.2 p.m.y. to research on chemical composition, physical properties and autoxidation and 0.5 to research on new chemical derivatives.

It is estimated that industry is conducting research on animal fats to the extent of about 180 p.m.y. This is divided among the meat packing and rendering industry, 100 p.m.y. devoted largely to applied research; chemical companies, 50 p.m.y. devoted largely to applied research and development; detergent manufacturers, 10 p.m.y. devoted largely to production problems; research institutes and foundations, 20 p.m.y. in research largely in the field of biochemistry and nutrition.

PROGRESS

A. Chemical Composition and Physical Properties; Autoxidation

1. Fractionation and Analysis of Lipids. More exact knowledge of the composition of animal fats, particularly with respect to the less common components, is of paramount importance in their utilization. The development of chromatography and other newer techniques has made it possible to separate many of the components of fats that was not possible by the older chemical methods. In conduct of this work, constant effort is being made to improve the analytical techniques so they might be applied to further separation and determination of fatty constituents.

These advanced analytical techniques have been applied to characterizing the fats in a number of products. Studies of the lipid components of hides involving the stratigraphic analysis of hide substance have shown the presence of unusual fatty acids containing odd-numbered (C_{13} and C_{23}), low (C_{12}, C_{14}) and high (C_{24}) monounsaturated acids. Wax esters, found chiefly in the outer layers, consist of sterol esters and esters of long-chain alcohols. (Studies on hydrogenation of fats have shown that certain hydrogenation catalysts did not produce preferential hydrogenation of polyunsaturated acids over the monosaturated acids, nor produce trans double bonds.) In studies on lard composition some 29 fatty acids have been identified as constituents of lard, and quantitative determination of 26 made. Studies on toxic chicken fat showed the presence of large amounts of conjugated linoleic acids compared with trace amounts in non toxic feed. A quantitative estimation of the fatty acid distribution between normal and high stearic acid fed rabbits has been made. A high stearic acid content of the diet was not reflected by a large increase in the stearic acid content of the rabbit fatty tissue. Thin layer chromatography has become an increasingly useful tool for many lipid studies. Improvements made in techniques and accessories for gas-liquid chromatography and thin layer chromatography are important in the further development of these procedures as quantitative methods of analysis of fats and their chemical derivatives.

2. Composition Changes of Animal Fats in Foods. Earlier work carried out on the problem of autoxidation of fats using pure amino acids and fatty compounds in model systems has shown that histidine has a marked catalytic effect on the autoxidation of methyl linoleate at 20°C. with oxygen in the dark. Oxidation values corresponding to 44% oxidation of the ester in the presence of histidine compares with 2.5% conversion when no histidine was present. Other amino acids did not have this effect.

Studies on this problem have been pursued further, determining the effect of other factors such as metal ions, pH, and emulsifiers, on rate of oxidation. The following progress has been made in these studies: (1) histidine is a powerful pro-oxidant for emulsified linoleate but not for dry linoleate, the pro-oxidant effect being enhanced many-fold by the presence of ionic iron, (2) ferrous and ferric ions are more powerful pro-oxidants than cupric ions in emulsified linoleate, either alone or with added histidine, (3) the pH effect is important, maximum pro-oxidant effect occurring at pH 6.5, and (4) the pro-oxidant effect of histidine can be inhibited with phosphate.

Further studies on the effect of the relationship of concentration of histidine to Cu^{++} and Fe^{++} ions in inhibiting autoxidation of fats reveal a rather complex picture. For example, in a 1:1 molar ratio of histidine to Cu^{++}, the histidine increased the pro-oxidant effect of Cu^{++} on linoleate emulsions, whereas in molar ratios of 2:1 the reverse effect was noted. In contrast, the pro-oxidant effect of Fe^{+++} is enhanced by histidine only when the ratio of histidine to Fe^{+++} is relatively high. At low molar ratios, the pro-oxidant action of Fe^{+++} is inhibited. These findings might be used to explain the batch-to-batch variation in such fatty foods as whole milk. In such a complex and delicately balanced system as milk, slight variations in the ratios of certain components might either enhance fat stability or decrease it.

The findings that non-ionic emulsifiers and phosphate do not support the pro-oxidant action of iron and histidine in the oxidation of emulsified linoleate may lead to questions on the validity of many previous autoxidation studies in which non-ionic emulsifiers have been used. From the results it would not appear justifiable to apply information obtained with anhydrous fats to preserving fat stability in foods containing water, or to emulsion polymerization of unsaturated organic monomers without first making studies in aqueous systems.

3. Synthesis and Physical Properties of Pure Triglycerides. Investigations on the synthesis and determination of structure and properties of pure triglycerides are being carried out through "in-house" research and through contract research with Villanova University. The "in-house" research has as its objective obtaining basic knowledge of the physical and chemical properties of the individual mixed glycerides of animal fat and how these properties are affected by the degree of unsaturation of the component fatty acids and by the relative position of these fatty acids in the glyceride molecule. The contract research aims to determine the molecular configuration of a simple saturated triglyceride through X-ray diffraction of a single crystal with a view to utilizing the basic information obtained in modifying fats to obtain specific physical characteristics in compositions in which they are employed.

The "in-house" phase of these studies has been completed. Methods have been developed for preparing and characterizing a variety of glycerides. The basic knowledge of the structure and behavior of pure glycerides accumulated under this line of study is necessary in a long range program of research aimed at developing new and improved products from animal fats. This type of investigation is being continued under a new project dealing with the structure of derivatives and components of animal fats employing a diversity of physical instrumentation. Investigations are being carried out on the structural features of closely related esters of fatty acids using low temperature absorption spectroscopy in the infrared region. New and improved methods for structure determination are needed as a basis for research on industrial utilization of chemical derivatives of fats.

B. Improved Polymers, Plastics, Resins and Lubricants

1. Resins. Two accomplishments have resulted from work carried out on investigations on resinous materials from animal fats: (1) preparation of polymers of vinyl stearate which are potentially useful as synthetic fibers with good water resistance, and (2) a general method has been devised for preparing amides from esters at a low temperature, which has attracted considerable industrial interest. Industrial development of these processes would serve as an outlet for substantial quantities of inedible animal fats.

2. Plastics. Since the plastics and plasticizer fields offer large potential outlets for chemical intermediates derived from animal fats, a variety of chemical components and derivatives of animal fats are being investigated for use in these fields.

(1) Vinyl copolymers from animal fats have been studied under a contract with the University of Illinois, supported in part by EU, and these studies are being continued under a contract with the University of Arizona, also supported in part by EU. Much basic information was developed in this work applicable to the preparation of vinyl polymers from animal fats, which has been reported on in 16 papers and a report on the conversion of agricultural products to polymeric materials, which makes possible the preparation of a wide variety of new copolymers of vinyl monomers with assured consistency of behavior. An extension of the work carried out at the University of Illinois is being carried on at the University of Arizona. Polymers derived from such animal fats derivatives as methyl and vinyl oleate and phenyl stearic acid are being prepared. Studies are also being carried out on the homopolymerization of vinyl dichlorostearate and the copolymerization of vinyl dichlorostearate and vinyl chloride. The copolymers have viscosities ranging from .7 to .9 and softening points between 50 and 100°. Polymers and copolymers of vinyl tetrachlorostearate also have been made. The results of this

work contribute to an assessment of the value of these fat-based monomers in the plastics industry.

(2) Octadecyl acrylate polymers have been investigated further. Attempts to improve the impact resistance of copolymers of octadecyl acrylate by copolymerizing with styrene have resulted in impact resistance equivalent to the best high-impact commercial polystyrene. The octadecyl acrylate polymers have properties that make them promising for industrial outlet. They are hard, tough, high-melting waxes that might serve as substitutes for expensive imported carnauba wax. Early attempts at copolymerization indicate that modification of commercial polymers with polymerizable amides may result in useful materials. Further studies have been carried out on copolymerization of N-allylstearamide and, respectively, acrylonitrile, vinylidene chloride and vinyl acetate. The preparation of copolymers of N-allylstearamide, a monomer readily prepared from animal fats, opens a new field of application of fat derivatives in polymer work.

(3) Studies have been continued on the preparation and reactions of glycidyl esters for use in the preparation of plastic resins. Additional diglycidyl esters have been prepared as part of the program to study the relationship between the structure of the ester and the physical properties of the corresponding resin. The high reactivity of glycidyl esters toward a variety of functional groups indicates the preparation of a variety of polymers will be possible. Evaluation of polymers resulting from diglycidyl esters and various acid anhydrides has indicated a number of interesting physical properties which probably can be improved under optimum curing conditions. Diglycidyl esters derived from fats appear promising as monomers for new resins and as comonomers for modification of existing resins. Hydration of the esters indicates that epoxidized esters can be converted to polyhydroxylated esters in high yield. The successful conversion of glycidyl esters to either monoglycerides or diglycerides should prove an inexpensive route to a variety of polyhydroxylated fatty acid esters.

(4) In studies on the preparation and polymerization of polymerizable amides from animal fats, monomer reactivity ratios of allylstearamide with several commercial comonomers have been determined. Conversion of monomer to polymer shows an unusual high dependence on the catalyst concentration, and the polymer has a typical allylic low molecular weight of 3000 to 5000. The variety of copolymerization possibilities demonstrated by copolymerization parameters for allyl stearamide indicates that some commercially successful copolymer systems might be found.

(5) Studies of preparation and reactions of organic-inorganic compounds from fats for use as plastic intermediates have been made. Investigations of the reaction variables and purification techniques were made in connection with dichlorocarbene addition and

etherification reactions to fatty compounds. The dichlorocyclopropanes have compatibilities with polymeric silicone oils. The etherification method affords fatty derivatives with added alkoxy, polyethenoxy, and ester functions of interest in several fields of application.

3. Lubricants. The field of lubricants and lubricant adjuncts is a very active one and offers appreciable potential for animal fat-based compounds. Synthesis of potential lubricants and their evaluation as lubricant additives have been carried out. Compounds have been prepared from a series of alkyl esters of 9(10)-bromostearic and dialkyl malonates by means of the malonic ester synthesis. Phosphonates also have been prepared from lard and oleo oil, and polycarboxylic acids and esters by condensing diethyl succinate and carbonyl compounds. Evaluation of the phosphorus-containing compounds shows very promising results. The materials appear to be effective as "anti-wear" and "EP" (extreme pressure) additives for petroleum lubricating oils and di-2-ethylhexyl sebacate lubricant base.

C. Improved Synthetic Detergents

1. Nonionic Derivatives. Surface active agents have been prepared by the reaction of ethylene oxide with 9,10-octadecandiol and with 9,10-dihydroxystearonitrile. The type of usefulness of such surface active agents appears to depend on the number of ethenoxy groups introduced into the molecule, the smaller number producing the better emulsifying properties. Oxyethylated 9,10-octadecanediols containing 4 oxyethyl groups are excellent emulsifying agents; those with 12 were good wetting agents. The nonionic soaps resemble noionics in general. Soap-like characteristics in foaming and detergency were overshadowed by nonionic characteristics. The products are comparable with other nonionics, but foaming, wetting, detergent and emulsifying properties are primarily dependent on the number of oxyethyl groups.

2. Soap-Detergent Combinations. Soap-detergent combinations based on animal fats have possibilities in both household and industrial applications. Certain low or controlled sudsing detergents and soap-detergent toilet bars appear to be the only present examples, and a systematic examination should reveal many other types of utilization possibilities. Based on our current investigations, products derived from the α-sulfonation of stearic, palmitic and pelargonic acids appear to be a promising type that may have detergent, lime soap dispersing, wetting, foam-stabilizing or emulsifying properties, depending on the particular structure. The α-sulfonation of pelargonic acid and esterification has led to the discovery of a new type of wetting agent. The α-sulfonation of the lower molecular weight fatty acids of cocoanut oil and of lauric and myristic acids, followed by esterification, also produced efficient wetting agents.

The best of this general class have wetting and foaming properties in hard and soft water equal to or better than the most efficient commerical wetting agents, with the added advantage that they are not easily hydrolyzed in hot acid or alkaline solution. Emphasis in the work has been placed on preparation of new derivatives and types of usefulness in combinations with soaps or other types of industrial surface-active agents. Symmetrical esters are excellent wetting agents. Diesters, diamides, α-sulfo tallow alcohols, and sodium alkanesulfonates are new types of derivatives that have increased the potential of α-sulfo tallow acids as products of commercial importance. Future work will be directed to studies of detergent formulations, soap-detergent combinations, and correlation of structure with useful properties.

D. New Chemical Derivatives

1. Addition of Nucleophiles. This work has emphasized the direct carboxylation of unsaturated fatty acids, esters and alcohols; the preparation of the dicarboxylic acids; and the controlled preparation of mono- and diesters of carboxystearic acid. The addition of formaldehyde to the double bond of fatty acids produces new types of useful compounds not hitherto available for study. The reactions give high yields and the products should have wide applicability in areas such as polymers, lubricants, plasticizers and functional fluids. The monoesters of carboxystearic acid are low-melting saturated acids hitherto unavailable. The preparation of 1,3-glycol and m-dioxane in high yield by addition of formaldehyde to oleic acid should provide compounds of wide utility as polymers, plasticizers, lubricants and other types of industrial products. Further studies are required on developing effective methods of separating the glycols from the dioxanes, so their physical and chemical characteristics can be determined.

Addition reactions of this type represent a fruitful field of exploratory research, as they offer a means of preparing new compounds not available through other methods of preparation. After preparation, these compounds must be evaluated for specific uses, which is carried out by a physical evaluation group after the compounds are prepared and purified.

2. Sulfur Derivatives. Studies on the preparation of sulfur derivatives of fatty components for industrial use have been completed. Synthesis of sulfonic acids with known position has been successfully accomplished through the thiolacetic acid addition-oxidation procedure. These compounds have been difficult to synthesize previously, and they will be evaluated for use in various applications.

3. Epoxidized and Hydroxylated Fat Derivatives. Work in this area involves preparation of long-chain products from oxidized (epoxidized

and hydroxylated) fat derivatives for use in polymer modification. Isopropenyl esters of stearic and oleic acids have been prepared by reaction of isopropenyl acetate with the fatty acid in presence of an acid. Further chemical transformation of the esters yields geminal diesters (2,2-distearoyloxy- and 2,2-dioleyloxypropane) and by analogous procedures 1,1-distearoyloxyethane and 1,1-distearoyloxycyclohexane. An interesting by-product in some of these reactions is the fatty acid anhydride. This reaction offers an opportunity to obtain some hitherto unavailable anhydrides by reacting fatty acids with isopropenyl acetate. These compounds being prepared are new and in some cases represent a class of compound not available before. When preparation of the series is complete their physical properties will be determined and they will be evaluated for various applications.

Attempts were made to prepare acylons through reductive debromination of a bromoketone acyl. The anticipated compound was not formed, but instead ethyl stearate or hydroxyacetone monostearate was produced depending on reaction conditions. A straight chain vicinal diketone was produced when methyl stearate was condensed with tetramethyl cyclobutanedione.

Studies on use of isopropenyl esters as acylating agents have been initiated. Isopropenyl stearate reacts with succinimide or phthalimide (acid catalyst) to form N-stearoylsuccinimide or N-stearoylphthalimide, respectively. Secondary amines can be similarly stearoylated. Hydrogenolysis of N-stearoylsuccinimide under mild conditions yields octadecanal, octadecanol, and regenerates succinimide. Acylation of nitrogen-containing compounds with active hydrogen atoms opens up an entirely new and broad field of fat chemistry. Acylation of barbituric acid and hydantoin derivatives should permit preparation of pharmaceutically useful materials. Acylation of polyhydroxylic substances (cotton, wool, starch, etc.) should yield substances of widespread importance and use.

4. Phosphorus, Sulfur, Oxygen and Nitrogen-Containing Fat Derivatives for Plastic Intermediates. This area of exploratory research deals with the synthesis of phosphorus, sulfur, oxygen and nitrogen-containing compounds from animal fats for use as plastic intermediates. During the reporting period work was carried out on phosphorus and sulfur compounds, as well as peroxides.

Relatively few synthetic compounds containing more than one phosphorus atom have been prepared and methods for preparing them are quite complex. Fat derivatives containing two phosphorus groups were prepared readily by addition of dialkyl phosphonates to diallyl esters of dibasic acids. These derivatives have not been previously reported. They are thermally stable, high-boiling liquid which will be evaluated as functional fluids, as lubricant additives and as

plasticizers. Since only one phosphorus atom in a high molecular weight plasticizer imparts some flameproofing characteristics to the resulting composition, it is hoped that esters with two phosphorus atoms will be outstanding in this regard. The addition of dialkyl phosphonates to linoleates and to unsaturated natural triglycerides such as soybean and cottonseed oils is a marked advance in this type of synthesis, since compounds containing relatively high phosphorus contents can be obtained in good yield from readily available inexpensive intermediates.

Studies have also been carried out on the direct production of peroxyacids from carboxylic acids and hydrogen peroxide in methanesulfonic acid solution. This reaction has been shown to be a fairly general one, and for the first time aromatic peroxyacids can be obtained directly. Studies on the direct synthesis of sulfonic acids from olefins by conversion to thiolacetates and oxidation have been completed. Isomer-free sulfonic acids from aliphatic compounds hitherto have been unavailable, and the availability of such compounds is necessary for determination of their properties and utility for industrial applications.

5. Dielectric Properties of Fatty Peroxides. This research being carried out under a contract project with the University of Pennsylvania has as its purpose studies on dielectric and related properties of fatty peroxides to obtain basic information for use in developing new products from animal fats. Investigations have been carried out in three physico-chemical areas: dielectric properties of t-butyl peresters; heats of combustion of peracids, t-butyl peresters and their corresponding non-peroxygen acids and esters; and kinetic studies on the decomposition of t-butyl peresters. Collectively, these studies will provide information on the structure of these peroxide derivatives and important thermodynamic data which will serve as aids in the development of reactions for converting peroxides of fats and their derivatives to more useful chemicals. Recent studies under the contract have shown that certain organic peroxides, notably diacyl peroxides and peroxy acids, can be decomposed to obtain useful and difficult to obtain long-chain compounds. Studies have been directed to increased yields of hydroperoxides, which are based on attachment of a group to the alkyl methanesulfonates, which increases their solubility and reactivity.

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AREA NO. 5 HIDES, SKINS AND LEATHER - PROCESSING AND PRODUCTS

Problem. To maintain the utilization of animal hides and skins at a profitable level there is need to find new products and processes to provide outlets for about 7 million cattlehides that are now available in excess of domestic needs. The foreign markets that currently absorb these surplus hides are also threatened by the increased hide production and decreased per capita use of leather (the principal outlet for hides) that have dislocated U. S. markets and caused prices to drop so precipitously in the last 10 years. To meet this problem there is need for upgrading the quality of raw hides and skins, for reducing the costs of producing leather, and for developing products with improved properties. To achieve these objectives research is needed to develop improved curing processes and agents, more effective control measures for (ante mortem) defects such as grubs, brands and parasite damage, and improved methods of take-off. Fundamental research is needed on the composition of hides to provide basic information on the chemical, physical and physical-chemical properties and reactions of collagen and other hide components for use in studies on chemical modification and on the development of new and improved products and processes. Development of new, more rapid and economic processes for curing, handling, unhairing and tanning hides is needed to reduce the cost of producing leather. There is also need for research on the chemical modification of hide proteins to develop leather products with such improved "built-in" properties as increased resistance to wear, scuffing and deterioration from perspiration, enhanced washability, dry-cleanability and improved dyeability; and to improve the physical and chemical properties of gelatin and glue, the important outlets for by-products.

USDA PROGRAM

The Department is conducting a broad program of basic and applied research on hides, skins and leather at Wyndmoor, Pennsylvania; Philadelphia, Pennsylvania; Lowell, Massachusetts; Kansas City, Missouri; and Cincinnati, Ohio; which involves chemists, biochemists, microbiologists, and leather technologists.

The Federal scientific effort devoted to the overall program totals 21.3 professional man-years, as follows:

(a) Chemical and physical properties and structure of hides involves 3.0 p.m.y. at Wyndmoor. This research involves exploratory research on the isolation and characterization of cattlehide components and the organization of the components within the hide structure and their relationship to leather properties. A contract with Lowell

Technical Institute Research Foundation at Lowell, Massachusetts, in the amount of 0.3 professional man year deals with the composition and properties of basement membrane of calf skin. A contract with the Franklin Institute, Philadelphia, Pennsylvania, in the amount of 0.7 professional man-year deals with the development of dynamic testing of leather. A contract with the University of Cincinnati, Cincinnati, Ohio, in the amount of 0.3 professional man-year involves basic studies on the noncollagenous proteins of cattlehides. Studies on the stratigraphic composition of hides was completed during the reporting period.

(b) Chemistry of collagen involves 4.0 professional man-years at Wyndmoor, and concerns determination of the physical-chemical properties of collagen, its soluble components and reaction products with modifying chemical agents, to elucidate the nature of the forces that control the stability and reactivity of this natural polymer and the factors responsible for the unique physical properties of leather. A contract with the Midwest Research Institute, Kansas City, Missouri, utilizing 0.5 professional man-year deals with the physical and chemical properties of collagen.

Additional research sponsored by the Department under grants of P.L. 480 funds is in progress at:

(1) University of Turku, Finland, (0.6 p.m.y.) for research on the fractionation of gelatin and soluble collagen.

(2) Central Leather Research Institute, Madras, India, (0.3 p.m.y) on the reaction of polyphenolic tanning compounds with hide proteins (collagen).

(c) Chemical modification of hides involves 6.0 p.m.y. at Wyndmoor. The program is aimed at developing new products with built-in properties tailored to specific end use requirements.

(d) New and improved processing involves 3.8 professional man-years at Wyndmoor. This research deals with new techniques for unhairing hides and skins, processes for imparting improved resistance of leather to deterioration by heat and perspiration, and new and improved tanning procedures.

A contract with the Lowell Technical Institute Research Foundation, Lowell, Massachusetts, in the amount of 0.3 professional man-year deals with the processing of leather tanned with dialdehyde starch.

Additional research is in progress under grants of P.L. 480 funds to the following foreign institutions:

(1) British Leather Manufacturers Association, Surrey, Great

Britain, (1.0 p.m.y.) for a study of deterioration of leather by sweat, chemicals and heat.

(2) Experiment Station for Leather Products, Naples, Italy, for research on the cause of "red heat" (0.7 professional man-year) and on improved tanning methods for United States hides (0.8 professional man-year).

(3) Central Leather Research Institute, Madras, India, (0.2 professional man-year) on the interrelation of hide quality with the rate of tanning and efficiency of tanning.

Studies on development of improved enzyme unhairing treatment for hides and the evaluation of dialdehyde tanned leathers were completed during this reporting period.

(e) Enzymology of hide processing involves 2.0 professional man-years at Wyndmoor, on studies on screening of commercially available enzyme preparations for their depilatory activity on cattle hides and skins.

A contract with the University of Cincinnati, Cincinnati, Ohio, in the amount of 0.5 professional man-year deals with the enzymic process of unhairing hides.

(f) Utilization of animal protein residues involves research in cooperation with the National Renderers Association, who support one professional worker for research on renderers protein residues (meat and bone meal and tankage) to obtain information for use in developing the best values from the residues. U. S. Department of Agriculture participation in the program is 0.2 professional man-year in a supervisory and research leadership capacity.

RELATED PROGRAMS OF STATE EXPERIMENT STATIONS AND INDUSTRY

State Experiment Stations in 1961 reported no work in this area.

Industry and other organizations are carrying out a valuable research program. It is estimated that 20 professional man-years are expended by a number of chemical companies who specialize in tannery and packing house supplies, for research on developing new tanning agents, unhairing agents, and the various chemicals used in the tanning and finishing of leather. About 10 professional man-years are expended by the tanning industry for developing new processing methods and techniques used in the tanning industry. It is estimated that 10 professional man-years per year are expended by universities and institutes conducting basic research on the composition and physical structure of hides and skins, on the chemistry of collagen, on the utilization of animal by-products and on improving the chrome tanning process. About 5 professional man-years per year are expended by the

meat-packing companies devoted to new and improved processes for handling and curing hides.

It is estimated that 5 professional man-years per year are expended by research and development corporations for research underwritten by segments of the meat-packing and tanning industries. This research includes projects such as developing a solvent tanning process for sole leather; a process for reconstituting hides; a scuff-resistant finish for shoe upper leather and conversion of tannery by-products to a feed stuff supplement.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE RESEARCH

A. Chemical and physical properties and structure of hides

1. Distribution and properties of hide components. A better understanding of the nature and distribution of hide components is essential to the development of improved methods of processing hides into leather and to the development of new and improved types of leather. Previous studies have shown that the density of the grain layer is less than the middle portion of the hide and contains considerably more void space than the center layers. Studies have also been carried out on stratigraphic distribution of the fatty components through the thickness of the hide which showed a considerable variation in the amount and nature of the fatty components. These results, obtained through use of a microtome, were based on layers which were only a few square centimeters in area. Bandknife splitting has been used to obtain much larger areas of the split hide, which has greatly facilitated the studies. A technique has been worked out for measuring accurately the void spaces in the hide caused by removal of the hair shaft. Results show that hair shaft volume is only a small percent of the total void space.

The studies on stratigraphic composition of hides for the most part have been completed. Determinations have been made of the distribution of dry matter, nitrogen, hydroxyproline, carbohydrates, hexosamine and lipids through the thickness of the hide. A marked variation in lipid content from layer to layer was found. It was also established that there is a lower density in both the grain and flesh regions of hides and that this density difference can be varied by physical treatments. The results indicate the plausibility of producing improved properties in leather by physical treatment.

2. Composition of the basement membrane of hides. Fundamental studies conducted under a research contract by the Lowell Technological Institute on the composition of the basement membrane of hides have shown that the only recognizable constituents present were elastin fibers and reticular-like fibers which appear to be continuous with similar fibers in adjacent tissue, and mucoproteins which were

concentrated in the basement membrane and did not appear to be present to any extent in adjacent tissue. The mucoprotein nature of the matrix explains the effectiveness of proteolytic enzymes as depilatory agents. It appears that the most efficient enzyme would have specificity for the protein component of the mucoprotein fraction of the basement membrane.

3. Reversible shrinkage in leather. Certain tanning materials impart the important property of reversible shrinkage to leather. For example, an epoxy resin (Epon 562) under certain conditions produces leathers showing reversible shrinkage. This property is important in imparting special properties to leather that could lead to expanding its use in applications such as leather garments and gloves. Studies to elucidate the mechanism of reversible shrinkage have developed information on the nature of the reaction between the epoxy resin and collagen molecules. The results indicate lysine, tyrosine and hydroxylysine have formed a stable combination with the resin. Since these amino acids are not terminal to the collagen molecule, the α-amino nitrogen could not be the point of attachment. The disappearance of certain amino acids from hydrolysates of epoxy-resin treated collagen suggests that they are involved in the reaction. The relatively strong chemical bond between the epoxy resin and amino acid is substantiated by the failure to detect the products using the usual tests for amino acids. Since the epoxy resin has 3 reactive groups, the possible complex nature of the derivatives is quite evident. Quantitative studies indicate that the equivalents of permanently attached resin are a simple multiple of the total resin content.

Identification of the structure produced by the reaction of collagen with the epoxy resin should throw considerable light on the mechanism responsible for the phenomenon of reversible shrinkage. An understanding of the true nature of the phenomenon might enable production of more economical and practical washable leathers.

4. Dynamic mechanical tester. A dynamic mechanical apparatus for use in evaluating the fundamental physical behavior of hide and leather is a valuable tool for obtaining basic information essential to progress in better utilization of hide substance. A research contract with the Franklin Institute of Philadelphia to design and construct an apparatus of this kind has been made, and work is under way. Good progress has been made, and the system proposed by the contractor appears to be highly practical.

B. Chemistry of collagen

1. Effect of electrolytes on collagen and its solubilization. Studies on collagen being carried out concern the determination of its chemical and physical behavior and the effect of electrolytes on its properties. It has been demonstrated that calcium ions are preferentially absorbed by collagen. A new form of collagen was

also produced to demonstrate that the structure of the collagen fiber depends on its previous environment. Preparations of soluble collagen have been made in a variety of buffer (salt) solutions and studied by means of ultracentrifugation and electrophoresis. Preparations made below a critical pH showed a single sharp sedimentation peak. Preparations of soluble collagen made with phosphate buffers were assayed to determine bound phosphate, and equilibrium-dialysis studies carried out using hide that had been subjected to washing prior to solubilization of the collagen. This was intended to remove polysaccharide hide components. The results showed that the presence of polysaccharides has no effect on the results. The fundamental information resulting from these studies will provide a basis for further studies on the solubilization, modification and stabilization of hide components.

In contract research at the Midwest Research Institute, an interesting and possibly quite significant new method for dispersing collagen was discovered. By forming the hypochlorite or hypobromite, in the absence of excess acid, the derivative disperses completely to give a gel containing as much as 3% collagen. Some degradation takes place, but collagen fibers can be regenerated.

Fundamental studies on the fractionation of gelatin and soluble collagen proteins and characterization of the fractions by chemical and physico-chemical means to provide information for expanding the utilization of hides and skins are being conducted under a P.L. 480 grant at the University of Turku, Turku, Finland. The grantee has investigated the fractionation of gelatins, using ion-exchange methods, and some of the techniques explored appear worthy of further study. These are being applied to a number of samples supplied him by the Eastern Utilization Research and Development Division of the Agricultural Research Service. Studies on the thermal and mechanical properties of rat tail collagen have revealed that the form of the tension-time curve of tendon fibers is changed within a few minutes after its extraction from the tail, and there are indications that the mechanical properties are influenced by the physiological states of the rat. The increased solubility and decreased tensile strength of rat tail collagen do not appear to depend on the amount of carbohydrate tissue. However, carbohydrates could form a protective coating on the surface of the fibers. Differences in the physical and chemical properties are being characterized by X-ray diffraction, infrared spectra, water vapor sorption analysis, binding power for chromium salts, rate of gelation and properties of the derived gelatins. The experimental design is such that the studies can be extended to reveal the tensile strength of recently formed interwoven collagen as a function of associated tissue components. The fundamental information being developed in this work should be helpful to a better understanding of the structure and behavior of collagen, which may have application to developing

improved leather and hide products.

C. Chemical modification of hides

1. Improved water repellency for leather. Imparting better water resistance to leather is important in improving its competitive position with respect to leather substitutes. Previous work has shown that silicone is effective for this purpose and that a silicone-tung oil mixture is essentially as effective as tung oil alone, which would reduce the cost of the process. Further studies confirm this water-proofing effect. However, the water-proofing agent is incompatible with conventional lubricating agents such as fat liquors, and studies being carried out to overcome this difficulty appear promising. Use of alkenyl succinic anhydride (Casyl B18) dissolved in mineral spirits as the lubricating agent is effective for glutaraldehyde-retanned chrome leather. A low level of water repellent treatment appears to be quite effective and offers promise for an economical use of silicone. The finding that retanning chrome leather with glutaraldehyde greatly simplifies the lubrication step has improved the potentialities for the tung-silicone complex as an economical means of imparting water resistance to leather. Water-repellency tests are being conducted with full sides of chrome-tanned splits which were retanned with glutaraldehyde and fat-liquored with Casyl B18.

Studies have been carried out on the use of urethane elastomers as impregnants for leather, especially chrome splits, in order to improve the resistance to water and perhaps to abrasion as well. Finished and rough sole leather and vegetable-retanned chrome splits were impregnated with urethanes, with and without a catalyst, in varying concentrations. Water resistance (static) was improved. Finished sole leather showed little change in abrasion, whereas rough sole leather and retanned chrome splits showed an increase in resistance to abrasion. Leathers impregnated with urethanes without catalyst were not as stiff and brittle as leather impregnated with urethanes with catalyst, and showed only slight differences in resistance to abrasion.

2. Chemical modification with dialdehydes. Dialdehydes have been shown to be versatile tanning agents and impart important properties to leather. Glutaraldehyde has been shown to be a good tanning agent for light leathers, either alone or as a combination tannage with chrome. Further studies have been carried out on the application of glutaraldehyde and dialdehyde starch as tanning agents. Studies on the rate of tanning with aldehydes and chrome used simultaneously were carried out. Comparison was made between formaldehyde and glutaraldehyde in such a combination. Both formaldehyde and glutaraldehyde appear to act independently of the chrome.

Skins tanned with glutaraldehyde and chrome were processed into finished garment leather in a commercial tannery. The finished leathers were of good quality. Tests showed a good degree of washability and resistance to a synthetic perspiration. Hot soap solution (wash test) lowered the shrinkage temperature of these leathers by 4 to 10°C. as compared with 22°C. for leather tanned with chrome alone. The glutaraldehyde-chrome leathers were resistant to area change embrittlement when heated in a synthetic perspiration in marked contrast to the behavior of chrome leather. It appears that a relatively small amount of glutaraldehyde is effective in producing these desirable properties in chrome leather.

Cooperative tests with tanners are being carried out to evaluate glutaraldehyde as well as dialdehyde starch in various combination tannages of grain split cowhide intended for upholstery and garment leather. The retention in leather of the desirable properties conferred by glutaraldehyde even when used simultaneously in the same bath with chrome is a step forward toward simplication of the process. This will give it the versatility necessary for ready adoption by tanners. Furthermore, the desirable properties of glutaraldehyde leathers can be conferred on the regular commercial chrome leather by merely adding a small percentage of glutaraldehyde to the normal tanning process. The versatility of glutaraldehyde allows its use along with chrome in the tanning of leather. The development of mellowness without loss of tightness has proved a most valuable property for the production of leathers for the currently popular softee and casual markets. Such leathers may be the answer to the threat posed by sneakers to the shoe upper leather market.

In contract research carried out by the Lowell Technological Institute on production of light leather with dialdehyde starch it was found that the use of ammonia during tanning with dialdehyde starch offers promise of developing suitable posttanning processes for leather tanned with dialdehyde starch along. A satisfactory procedure has been developed for tanning garment type leather. This involves pretanning the skin with a small amount of chrome (2%) followed by retanning with dialdehyde starch. These leathers are almost white and can be readily rewet for staking. The staked leathers are smooth-grained and soft.

D. New and improved processing

1. Imparting improved deterioration resistance to leather. Leather with greater durability is desired for most applications. Perspiration resistance is one of the properties that needs to be improved. Department research in the past has developed greater perspiration resistance to insole leather. Present efforts are being directed to improving light leathers in this respect, such as glove, garment and shoe upper leathers.

Work has been continued on the use of dialdehyde starch (DAS), glutaraldehyde and tetrakis-(hydroxymethyl) phosphonium chloride (THPC) in various combinations with conventional tanning agents to improve perspiration resistance. Vegetable-tanned bellies retanned with a small amount of glutaraldehyde resulted in improved perspiration resistance. Pretanning sole leather with dialdehyde starch improves perspiration resistance. However, to take advantage of dialdehyde starch, the whole tanning procedure would have to be changed. It was found that dialdehyde starch can be used effectively by incorporating it in the oil wheel, which is the last step in processing. This permits treatment with dialdehyde starch of only selected lots as needed. Another process for conferring perspiration resistance is use of tetrakis-(hydroxymethyl) phosphonium chloride-resorcinol in combination with dialdehyde starch or glutaraldehyde. Skins tanned with tetrakis-(hydroxymethyl) phosphonium chloride-resorcinol and retanned with various commercial tanning materials and then processed into garment suede or grain leathers were found generally to be of acceptable commercial quality.

A fundamental study of the mechanism of deterioration of leather to obtain basic information for use in developing leathers more stable to the action of chemicals, sweat and heat has been in progress at the British Leather Manufacturers Research Association at Milton Park, Egham, Surrey, England, under a P.L. 480 grant. Results obtained show that lactic acid in perspiration is the main cause of damage to chrome leathers and that small amounts of vegetable tannins increased the stability of chrome leather to perspiration. Studies have also been made on the combined action of heat and moisture as a cause of leather deterioration. It was found that chrome-tanned leathers are likely to be more resistant, while semi-chrome and chrome retan leathers are generally badly damaged by exposure to moist heat. Results of studies to determine the mechanism of deterioration suggest that the polypeptides of the chains of the collagen are broken down to smaller units and there is general disorganization of the protein structure. The changes resulting from γ-irradiation of collagen are of a similar pattern, and this may throw light on the process involved.

Studies on stabilization of leather through cross-linking show that tanning by the introduction of cross links, such as is imparted by glutaraldehyde, reduces breakdown of the polypeptide chains and thus tends to stabilize the leather. This confirms observations made in the United States Department of Agriculture that dialdehydes, and in particular glutaraldehyde, are good cross-linking agents and tend to stabilize leather.

2. Enzymatic unhairing of hides and skins. The development of an economical process for enzyme unhairing of hides that would produce high quality leather would have a number of advantages, including

that of stream pollution abatement. When tanned by usual processes for shoe upper leather, the leather from enzyme-unhaired hides is flat, firm and tinny. Tests have been carried out with the object of producing commercially acceptable chrome-tanned leather from enzyme-unhaired stock. Postliming of hides for a day after unhairing produces leather of acceptable quality. This indicates that some physico-chemical change is required in the collagen fibers.

In contract research conducted at the University of Cincinnati, steer-hides unhaired by two different enzyme preparations were vegetable tanned in the usual rocker yard and processed into finished leather. the finished leather from the enzyme-unhaired stock could not be distinguished from the matched sides which were unhaired with lime-sulfide. Appearance and properties were not significantly different.

Production of vegetable-tanned sole leather from enzyme-unhaired hides without need for modifying the tanning process is encouraging. Production of acceptable chrome-tanned upper shoe leather has proven to be much more difficult. However, several facts have been found that may prove to be clues to a solution of the problem.

3. New tanning processes. Studies initiated recently are aimed at improving the quality of leather utilizing newly discovered tanning agents. Tannins being investigated include glutaraldehyde alone, aldocryl alone, combinations of these tannins with chrome, formaldehyde and acrolein. Properties of particular interest are finish and durability of shoe upper, suede, garment and other types of leather. Leathers with greater perspiration resistance and durability would be advantageous, particularly for shoe upper leather and work glove leather. Evidence has been obtained that glutaraldehyde increases the perspiration resistance of leather. Findings that use of glutaraldehyde for tanning cattlehides either alone or in combination with chrome is entirely practical from a tannery standpoint have stimulated several tanners to evaluate this tannage in their production. This development is most timely, since it will enable tanners to produce leathers needed for the casual and softee shoe trade.

4. Investigations on "red heat." A fundamental study of "red heat" of cattlehides to obtain basic information for use in developing means of preventing this defect in exported American hides has been initiated by the Stazione Sperimentale per l'Industria delle Pelli e delle Materje Concianti, Naples, Italy, under a P.L. 480 grant. Studies are directed to isolating and culturing microorganisms associated with red heat in hides. Over 200 strains were cultured and characteristics determined. Investigations are also being carried out on the extent of damage done to hides by growth of pigmented halophilic microorganisms and the use of chemical agents for inhibiting their development. This information will be helpful

for preventing or controlling damage to hides caused by micro-organisms during storage or transoceanic shipment.

5. Conversion of U. S. cattlehides into sole leather in Italian tanneries. Due to difficulties encountered in the conversion of U. S. cattlehides to high quality sole leather in Italian tanneries, research has been undertaken by the Stazione Sperimentale per l'Industria delle Pelli e delle Materie Concianti, Naples, Italy, into the cause of this behavior of American hides as compared with hides from other countries. They have found that American hides possess 10 to 20 times more fat than their domestic hides, and even after degreasing they still had 2 to 4 times as much.

Tanning tests on American hides tanned by Italian processes have been carried out and the leather compared with sole leather produced by using the English process. The grantee has completed the evaluation of U. S. Packer hides obtained from 13 sources. The tests confirmed previous results that U. S. hides have too many defects, such as fat, manure, dirt, grub holes and brands. On the basis of the pilot tanning tests, the nature of the problem of utilizing U. S. Packer hides in Italy and other European countries and the course of the investigation have been defined quite clearly.

E. Enzymology of hide processing

1. Screening commercial enzyme preparations for depilatory activity. Over 50 commercially available enzyme preparations have been screened for their depilatory activity in connection with enzymic removal of hair from hides. Several preparations of bacterial origin and one from a Streptomyces were found to be most active. The hair loosening activity was found to be associated with the proteolytic activity of the enzymes as demonstrated by action on casein, gelatin and elastin. Contrary to reports in the literature, no correlation was found between the amylolytic or elastolytic and depilatory activities of the enzyme preparations. The work carried out in this field has stimulated the interest of many tanners, packers and enzyme manufacturers to investigate commercial possibilities. No serious difficulties have been encountered in the use of conventional tannery equipment such as paddles and drums for unhairing operations. Studies on the screening of enzyme preparations have been completed.

2. Properties of leather made from enzyme unhaired hides. Contract research at the University of Cincinnati on the properties of enzyme-unhaired vegetable-tanned sole leather as compared with similar leather prepared from lime-sulfide unhaired hides has shown that finished leather from enzyme-unhaired stock was quite comparable to that prepared from lime-sulfide unhaired hides. Production of acceptable chrome-tanned shoe upper leather has proven

to be much more difficult. While the research has shown that the strength of enzyme-unhaired chrome-tanned upper leather is equal to that of similar leather made from hides unhaired by lime-sulfide treatment, the leather from the enzyme-unhaired hides was firmer and lacked the stretch characteristics required of shoe upper leather. The contractors feel that the firm tinny condition is a result of the nonuniform deposition of the chrome in the fiber. As a result of further systematic exploration of the problem, the number of variables that affect the mellowness and smoothness of grain in enzyme-unhaired stock is being reduced. The pickling process and the type of retannage appear to be most critical. Conditions have been found that produce the desired properties in laboratory size specimens. These should be confirmed using full sides.

F. Utilization of animal protein residues

Work on this project is being conducted through a fellowship arrangement with the National Renderes Association. The research was reactivated about a year ago when a new senior fellow, Dr. Gorbunoff-Timasheff, was appointed to continue the work. She will be assisted by a junior scientist. Work under the fellowship has been suspended for approximately two years because of resignation of both the former senior and junior fellows.

Work was resumed on amino-acid composition and fractionation studies of a number of samples of commercial meat meal. The meals were fractionated into homogeneous components suitable for further study. A method has been developed for separating the bone from the meat portion of the meal. Studies also indicate a considerable breakdown of the protein of the meat portion of the meal, which evidently takes place in the rendering. It is not known whether this breakdown of the protein affects its feed value. Studies being conducted by the National Renderers Association at Battelle Institute indicate that an enzymic rendering process they have developed may be more economical than the present one. This may produce meal with considerably less decomposition of some of the essential amino acids. The product made by this process would be completely soluble.

The fractionation process developed for separating the bone and meat fractions of meal will permit studies of the isolated protein without complication of mineral constituents. Information being obtained could lead to production of a more uniform product and thus help to stabilize the utilization of this valuable by-product of the rendering industry, which amounts to about 1,300,000 tons annually.

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* Work done under a P.L. 480 foreign research grant.

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AREA No. 6. POTATOES - PROCESSING AND PRODUCTS

Problem. The potato industry, faced with a continuing decline in the consumption of fresh potatoes, is becoming more and more dependent upon the development of new and improved processed products to maintain markets and to avoid recurring economic disasters. Crop perishability, supply fluctuations, and the inelasticity of demand, result in wide swings in price with even slight surpluses. In producing areas having a substantial processing industry, depressive lows are moderated by advance contracting by processors prior to harvest. However, in many important potato growing areas processing has not yet developed, and vulnerability not only still exists, but is exaggerated by the growing competition of processed potato and other competing food products. A continuing improvement in processed potato products is clearly required if processing is to expand fast enough to offset the progressive decline in use of fresh potatoes.

Lack of adequate knowledge concerning the chemical constituents, physical properties, and enzyme systems in potatoes is limiting development of new and improved processed products and processing methods. Basic research on composition is needed to provide fundamental information on which an applied research program can be systematically and effectively built. Recently developed techniques make it possible to isolate and characterize the constituents responsible for flavor, color, odor, and texture of many processed food products. Application of these techniques to potatoes and potato products should make it possible to improve the quality of present products, both freshly processed and following storage, and provide a basis for technological and engineering studies in new product development.

USDA PROGRAM - EASTERN REGION

The Department has a continuing long-term program of basic and applied chemical and engineering research on studies related to processing. The work of the EURDD, involving the services of chemists, biochemists, food technologists and chemical engineers at Wyndmoor, Pennsylvania is conducted in cooperation with the Maine Agricultural Experiment Station and several other stations, which supply potatoes of known cultural history. The chemical research program includes evaluation of the effects of variety, location of production, storage conditions and tuber solids content on potato composition with particular respect to nitrogenous constituents; principal acids and factors related to discolorations such as after-cooking discoloration. The Eastern Division's engineering and development research program seeks to improve the quality,

nutritive value and storage stability of dehydrated potato products and to develop more convenient types of dehydrated products, such as "instantized" slices that cook quickly.

The Federal (Eastern Division) scientific effort devoted to research in this area totals 13.0 professional man-years. Of this total, research on chemical composition as related to processing characteristics comprises 10.0 p.m.y. During the year, research at Wyndmoor, Pa. on potato after-cooking discoloration was terminated. Research on dehydrated potato products constitutes 2.0 p.m.y. During the year, research at Wyndmoor on development of potato flakes was completed. Research on new and improved processing technology amounts to 1.0 p.m.y.

RELATED PROGRAMS OF STATE EXPERIMENT STATIONS AND INDUSTRY

State Experiment Stations in 1961 reported a total of 7.5 professional man-years divided among subheadings as follows: Chemical composition and physical properties, 4.5; new and improved food products, 1.1; new and improved processing technology, 1.9. Relationships that exist between the total solids contents, groups of constituents and individual constituents and the various quality attributes of potatoes are being determined. Basic information is sought that will be useful in improving current products and processing techniques, with attention to color and flavor problems.

Industry and other organizations conduct a considerable amount of research on the composition and properties of potatoes and on development of new products and improved processes. Basic research is confined almost entirely to a few large firms, with applied research carried out by these large companies as well as by the smaller companies and the trade institutes. While liaison is maintained to promote the flow of nonconfidential information between the Department and industry, much of the industrial research findings are not disclosed to others because of commercial advantage attached to such information. Estimated annual expenditures are equivalent to approximately 55 professional man- years.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition as Related to Processing

1. Nitrogenous Constituents. Analysis of many potato samples has indicated, for the most part, that no spectacular or regular changes occur during the storage season in the amounts of the amino acids present. For a given production locality, nitrogen content on the fresh weight basis of potato is nearly constant from sample lot to sample lot. Maine and New York potatoes from the 1960 crop were first surveyed for amino acid content. This was followed by 1961

crop potatoes (Katahdin, Russet Burbank, Kennebec, Red Pontiac, and Cobbler varieties) from Maine, New York, Pennsylvania, Red River Valley area, Wisconsin and Idaho. Voluminous data have been collected from extracts run through the automatic amino acid analyzer. While computations have been made up to the present only manually, results will be obtainable much quicker when the necessary programming is completed to permit electronic computation of data from punched tape.

Exploratory work on isolating potato protein fractions has demonstrated that considerable effort will likely be required on modification of conventional methods.

Work is also in progress on the subject of sugar-amino acid compounds formation in potato chip frying but this has not progressed to the stage that findings can be reported.

2. After-cooking Discoloration. While after-cooking discoloration can be controlled by dipping sliced potatoes in a dilute solution of an acid such as citric or the salt sodium acid pyrophosphate, processors would like to be able to solve the problem through agronomical changes. A large number of samples of potatoes, representing various degrees of discoloration, were examined during the report period. Since the discoloration usually occurs more intensely at the stem end of the tuber than at the bud end, emphasis has been placed on differences in chemical composition of the two ends. After making determinations of a rather large number of constituents and groups suspected of being involved in after-cooking discoloration, three factors appear to be of importance: potassium content; iron content; concentration of acids.

The potassium content was less in every case in the stem than in the bud end. Statistical analysis of all potato samples examined with respect potassium content and extent of after-cooking discoloration showed an inverse relationship significant at the 1% level.

Increased blackening was correlated with increasing iron content. This was evident for both iron which is "free", that is not precipitated by heat coagulation of the protein in the juice, and for iron that is associated with the protein. A relatively high amount of the total iron is associated with the protein.

The bud end of the tuber has a higher acid content, which is consistent with the lesser blackening tendency there. Three to ten times as much malic, citric, orthophosphoric and oxalic acids are present in the bud as in the stem end.

3. Organic Acids. A study of the changes in the amounts of nonvolatile organic acids, occurring during 38° F. storage, was

completed in 1961. Knowledge of the extent of these changes, most rapid in the first two months of storage, is expected to be helpful in the development of relationships between potato composition and processing quality.

B. Dehydrated Potato Products

1. "Instantized" Pieces. It is believed that a great potential exists for dehydrated potato pieces that will rehydrate more rapidly than the conventional product. Methods have been developed for partially drying the pieces, rendering them porous by pressure build-up in a cereal gun followed by sudden release to atmospheric pressure, and finally drying the porous structure to low moisture content. Initial drying is carried out at somewhat above 120° F. A product of good uniformity in rehydration is obtained by "puffing" the pieces (3/8 inch cubes) at 40% moisture and in the pressure range of 55-65 p.s.i.g. "Instantized" dehydrated pieces will reconstitute in less than 5 minutes in boiling water as compared with 20-30 minutes for present commercial pieces. Cost estimates are now being prepared for projecting to large-scale operation.

2. Flakes and "Flakelets". Work on methods of producing potato flakes was completed in July 1961, with the satisfaction that it had resulted in present annual capacity to produce sixty million pounds of product, which is equivalent to 7 million bushels of potatoes yearly. Procedures were developed for producing high quality flakes by precooking potato slices, cooling, adding mono-glyceride emulsifier to provide "abuse tolerance", completing the cooking, and finally drum drying to give the end product. Through increased "abuse tolerance", it is possible to reduce the flake size and thereby increase the package density to 25-27 pounds per cubic foot while still retaining good texture on reconstitution. "Flakelets", having 45-50 pounds per cubic foot density, are produced by mixing the drum dried sheet, broken to about 1/4-inch size, with fresh mash of controlled moisture and then manipulating to achieve compaction and lamination. Flakelets are of interest to the QMC because of their high density, and of interest to industry because of the favorable estimate of cost to produce and the feasibility of using nitrogen packaging.

C. New and Improved Processing Technology

1. Potato Flakes and Flakelets Storage. Since the FDA has set a limit of 20 ppm of BHA plus BHT antioxidants in flakes, lower than the optimum for best storage stability, it has become advisable to search for other effective stabilizing agents. A flakelets storage test is now in progress in which mixed tocopherols and nitrogen pack are being compared with BHA plus BHT for effectiveness in stabilizing flavor of the product.

2. Technological Research in Processing. The project on technological research related to the potato processing industries was terminated in September 1961. This research resulted in important contributions including: development of streamlined methods for determining the fat content of chips and French fries and the SO_2 content of sulfited potatoes and flakes; determination of the factors governing the absorption of SO_2 by peeled, raw potatoes; utilization of the byproducts from chip and starch processing. It is anticipated that this area of work will be given further attention, with the development of a new line of research directed toward solution of current problems in the processing industries.

PUBLICATIONS AND PATENTS REPORTING RESULTS OF
USDA AND COOPERATIVE RESEARCH

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New and improved processing technology

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Kissinger, John C. 1961. A procedure for the bacteriological examination of potato flakes. U. S. Dept. Agr., Agr. Research Service Circ. ARS-73-32, 7 pp.

Ross, L. R. and Treadway, R. H. 1961. Factors affecting the sulfur dioxide uptake in sulfited pre-peeled potatoes. American Potato Journal, 38, pp. 9-13.

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AREA NO. 7. VEGETABLES - PROCESSING AND PRODUCTS - EASTERN REGION

Problem. Vegetables occupy over 3 million acres, with a yearly farm value of a billion dollars. Classic studies have revealed their gross composition (moisture, protein, carbohydrate, fat, minerals) but very little is known of the individual constituents that make up these broad classes, and still less about components outside these classes. This ignorance is a limiting factor in the development of new and improved processing methods and processed products, and technological advances have been hampered by insufficient knowledge of the constituents responsible for the color, flavor, and texture of vegetables, and the changes these constituents undergo during processing, storage, and distribution. There is need for basic compositional research to provide the fundamental information on which an applied research program can be developed logically and efficiently. Recently developed equipment and techniques have made it possible to isolate and characterize constituents that could not have been studied effectively with older procedures. There is also need for application of these results to developmental research on new products and new and improved processing technology.

USDA PROGRAM

The Department has a continuing long-term program employing chemists and chemical engineers in basic and applied research on vegetable processing and products. Research (EU) on inhibitors for pectinolytic and cellulolytic enzymes, extractable from plant sources such as grape leaves, is conducted at Wyndmoor, Pennsylvania. Pilot plant research on new, dehydrated vegetable products is also carried out at Wyndmoor. Research on new processing procedures to produce better quality canned vegetables has been conducted under contract at the New Jersey Agricultural Experiment Station, New Brunswick.

The Federal (EU) scientific effort assigned to this area totals 3.2 professional man-years. Of this total, research on chemical composition and physical properties constitutes 1.0 p.m.y. Research on new and improved dehydrated products comprises 1.0 p.m.y. Research on new and improved processing technology amounts to 1.2 p.m.y. During the year, contract research at Rutgers University, New Brunswick, N. J. on new processing procedures (high-temperature short-time) to produce better quality canned vegetables was terminated.

Related programs of all State Experiment Stations and industry and other organizations is reported by the Western Utilization Research and Development Division in that Division's Summary of Current Program and Preliminary Report of Progress.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Enzyme Inhibitors from Plant Sources. It has long been known that an inhibitor is present in Scuppernong grape leaves which prevents the enzymic softening of cucumbers during brining. Evidence has been adduced that a tannin is the factor which inhibits the action of pectinolytic and cellulolytic enzymes in softening cucumbers. A method has been developed for isolating the enzyme inhibitor from grape leaves and sericea; this procedure should be helpful to other investigators in plant science. The work should also be of value to processors in enabling them to consistently obtain firm cucumber pickles.

B. New and Improved Dehydrated Products

1. Puffed Dehydrated Vegetable Pieces. Methods have been developed for imparting a porous structure to partially-dehydrated vegetable pieces which speeds up the final dehydration of the slices and makes them much more readily rehydrated. Following the first stage of drying, the pieces are heated in a closed vessel (having a quick-opening lid) until the water contained within the piece is superheated with respect to steam at atmospheric pressure. The vessel is then instantly discharged to atmospheric pressure. The porous structure of the pieces is the result of the flashing of water vapor from all parts of the piece. While optimum conditions apparently vary somewhat from vegetable to vegetable, pieces should be dried to 25-50% moisture and then released about 35 p.s.i.g. for good results with 3/8 inch carrot and beet cubes. Promising preliminary results have also been obtained in the "explosive puffing" of peas, lima beans, and sweet corn kernels. Future work will be directed toward determining best conditions for "instantizing" a variety of vegetables while preserving original flavor and color to the maximum extent possible. Industry is keenly interested in the instantized vegetable pieces, which reconstitute in a matter of minutes. Cost estimates are being prepared.

C. New and Improved Processing Technology

1. High-Temperature Short-Time Canning. The objective of this research on high-temperature short-time (HTST) sterilization of canned Eastern vegetables was to evaluate anticipated quality improvement of HTST preserved products over those canned by conventional procedures. It is indicated that HTST processing yields higher quality canned green beans and tomato juice and minimizes textural changes relative to results obtained with the same products under conventional processing.

PUBLICATIONS REPORTING RESULTS OF USDA AND COOPERATIVE RESEARCH

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New and Improved Processing Technology

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Joffe, Frederick M., Stier, Elizabeth F., Bongolan, Dolores, Epstein, Arnold I. and Ball, C. Olin. 1961. Low-temperature handling of sterilized foods. IV. Color and flavor of canned vegetables. Food Technology, 15, pp. 507-510.

Joffe, Frederick M., Stier, Elizabeth F., Gustafson, Lanny L., Epstein, Arnold I., Hayakawa, Kan-ichi and Ball, C. Olin. 1961. Low-temperature handling of sterilized foods. VI. Color and flavor of canned soups, tomato juice, and evaporated milk. Food Technology, 15, pp. 510-514.

AREA NO. 8. APPLES AND OTHER FRUITS - PROCESSING AND PRODUCTS - EASTERN REGION

Problem. Lack of knowledge of the nature and quantities of the various chemical constituents and enzyme systems present in fresh fruits, and of the changes these undergo during processing, is a limiting factor in research designed to develop new and improved products and processing techniques. Knowledge is required on the composition and physical structure of fruits and fruit products, with emphasis on substances responsible for color and flavor, vitamins, and other constituents important in determining consumer acceptance and nutritive value of the products. Composition should be studied in relation to variety, stage of maturity, and environmental conditions of growth; and to changes occurring between harvesting and processing, during processing, and in storage and distribution. Recently developed equipment and techniques have made it possible to isolate, separate, and identify constituents that could not have been handled previously. As basic information is developed, new processing techniques will be applied in the improvement of fruit products, the development of new fruit products, and in more efficient utilization of by-products from fruit processing.

USDA PROGRAM

The Department has a continuing long-term program involving chemists, biochemists, and chemical engineers engaged in both basic and applied research related to extending the use of fruits in the food processing industries. In the EU program, apple products research, and investigations on the chemistry and cell structure of cherries are conducted at Wyndmoor, Pa. Development of rapidly-reconstitutable dehydrated fruit pieces is also underway at Wyndmoor. Contract research on peaches is in progress at Rutgers University, New Brunswick, and on apple texture at the Maryland Agricultural Experiment Station, College Park. The Federal (EU) scientific effort devoted to research in this area totals 9.7 professional man-years. Of this total, research on chemical composition and physical properties constitutes 6.0 p.m.y.

Research on new and improved food products amounts to 2.3 p.m.y. including 0.3 p.m.y. of contract research on apple texture at the Maryland Station. During the year, contract research on the use of fruit essences and concentrates in frozen desserts at the Maryland Station was terminated. Research on new and improved processing technology amounts to 1.4 p.m.y., including 0.4 p.m.y. of contract research on peach processing at Rutgers.

RELATED PROGRAMS OF STATE EXPERIMENT STATIONS AND INDUSTRY

Related programs of all State Experiment Stations and industry and

other organizations is reported by the Western Utilization Research and Development Division in that Division's Summary of Current Program and Preliminary Report of Progress.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties

1. Chemistry and Cell Structure of Cherries for Processing. Previous work on this subject has been concerned with (a) composition of cherries as affected by bruising and aging prior to canning and (b) influence of harvesting methods on quality of red tart cherries (in cooperation with the Agricultural Engineering Research Division). It was established that no sharp change in respiratory activity occurs as cherries mature, which means that harvesting may be spread over a relatively long period with very little physiological change or quality impairment. Some bruising inevitably occurs during cherry picking and handling. It has been found that the firming which takes place when raw cherries stand at orchard temperature for several hours between harvesting and canning is accompanied by conversion of a portion of the pectin to pectic acid by demethylation. The pectic acid may be responsible for the more rigid cell walls of the firmer fruit. An additional discovery was that techniques employing radioactive carbon 14 can be used to study the effects of harvesting and postharvesting practices on the fruit quality.

Mechanical harvesting of cherries has been practiced only for the past two seasons to any appreciable extent. This research has led to improvements in the type of shaker used and in decelerating the fall of the cherries into the catching net by use of 6-inch wide ribbons. Wide adoption of mechanical harvesting of cherries will involve changes in processing methods, preprocessing treatments, equipment and grade standards all of which will require research.

2. Influence of Carbohydrates in Apple Texture. This contract research at the Maryland Agricultural Experiment Station has been concerned with the relation between the insoluble carbohydrates and the texture of raw and processed apples. York Imperial, Stayman and Golden Delicious varieties were harvested at four stages of maturity and fruit was removed from storage at 25, 50, 75 and 100% of the expected storage life. Apple sauce and canned slices were prepared from each treatment. It is indicated that starch content decreases and cellulose increases at maturity. While it is not yet possible to conclude what is the optimum, it seems that the ratio of starch to other polysaccharides may be useful as a guide for deciding the time of harvest and storage time of apples intended for processing. The softening of apples during storage appears to be due to changes in a starch-pectin complex. Quality control in apple processing by carbohydrate analysis seems to be a fertile field for additional investigations.

B. New and Improved Food Products

1. Rapidly-Reconstitutable Dried Fruit Pieces. A porous, rapidly-reconstitutable structure is imparted to partially dried fruit pieces, e.g. 3/4-inch apple cubes, by heating them above 100° C. in a puffing gun and then suddenly releasing the pressure. Drying of the porous pieces to a low moisture-content is then readily carried out by ordinary methods. The "instantized" dried apple pieces can be reconstituted to a compote by boiling one minute in water containing sugar, or they may be mixed with dry cereal to contribute a crisp apple component. The "instantizing" process, which is attracting much outside interest, is to be applied to blueberries and to other fruits.

2. Improved Apple Cider. In studies on the preservation of cider by sorbic acid it was discovered that hydroxymethylfurfural (HMF) is formed during heat treatment of cider. Pasteurized cider darkens on storage, with accompanying increase in HMF and sedimentation. Model systems have been used to study the role of HMF under a wide range of conditions. Subsequent work will be concerned with improvement of cider quality and development of new cider products through knowledge gained with these model systems.

3. Dried Apple Sauce. Since dried apple sauce (now made commercially by drying apple slices, grinding and adding sugar) provides good color and texture in the reconstituted product, the Quartermaster Corps procures this item in volume. The current product, though, is notably lacking in characteristic apple flavor. A taste panel definitely preferred reconstituted sauce with essence containing apple juice powder in comparison to sauce reconstituted from the unmodified product. The Quartermaster Corps now has this improved product under test.

4. Essences and Concentrates in Frozen Desserts. Studies on the use of fruit essences and full-flavor concentrates in supplementing fresh fruit in ice cream and other frozen desserts showed that a significant flavor improvement could be realized without impairing the body and texture of the frozen dessert. Formulas and cost data are now being made available to ice cream manufacturers and efforts are being exerted to secure commercial adoption.

C. New and Improved Processing Technology

1. Processing Characteristics of Eastern Peaches. More than 300 new strains of peaches are available from the breeding plots at the New Jersey Agricultural Experiment Station. These are to be screened to appraise their potential value for processing. Promising strains will later be more rigorously evaluated by a panel for color, flavor, and texture. It is hoped that one or more Eastern varieties of peaches can be developed that will can well and possibly also be good for fresh use. Work on this project began in June 1962.

PUBLICATIONS AND PATENTS REPORTING RESULTS OF USDA AND COOPERATIVE RESEARCH

Chemical composition and physical properties

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Pollack, R. L., Hoban, N. and Hills, C. H. 1961. Respiratory activity of the red tart cherry (Prunus cerasus) during growth. Proceedings of the American Society for Horticultural Science, 78, pp. 86-95.

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New and improved food products

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Weaver, E. A., May 2, 1961. Preservation process. U. S. Patent No. 2,982,697.

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New and improved processing technology

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Gaston, H. P., Levin, J. H., Hedden, S. L. and Whittenberger, R. T. 1961. Experiences in mechanical harvesting of cherries. Michigan State Horticultural Society, Annual Report 90, pp. 14-25.

Harrington, W. O., Jahn, A. S. and Hills, C. H. 1962. Spectrophotometric determination of sorbic acid in apple cider. Journal of Food Science, 27, pp. 15-19.

Whittenberger, R. T. and Hills, C. H. 1960. Bruising of red cherries in relation to the method of harvest. U. S. Dept. Agr., Agr. Research Service Circ. ARS-73-27, 13 pp.

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AREA 9. TOBACCO - COMPOSITION AND PROCESSING

Problem. Although neither food nor fiber, tobacco nevertheless is grown on about a million acres, and in seven states provided more farm cash receipts than any other field crop in 1960. The farm value is about $1 billion. This crop is unique in that it yields about $3 billion in Federal and State taxes. Several serious difficulties plague the industry, among them the lack of genuine scientific knowledge of the composition of tobacco and tobacco smoke and the lack of objective standards of quality at all stages of processing. By knowing the chemical factors responsible for high quality it would become possible to predict accurately the usefulness of a particular tobacco for smoking purposes, and thus solve a long-standing industrial problem. Methods could also be devised to expedite current time-consuming and erratic methods of fermenting cigar tobacco. Finally, more selective studies on tobacco smoke could be made, including the origin and fate of leaf constituents during burning, the formation of substances having physiological effects, and ways of producing smoke of diverse composition.

USDA PROGRAM

The Department has a continuing program involving chemists and biochemists engaged in basic and applied studies of the chemical composition of tobacco leaf and smoke, directed to better understanding of and improvement in tobacco quality, and to improvement in tobacco processing technology.

The Federal work is conducted at Wyndmoor, Pa., and totals 7.0 professional man-years, 4.0 of which are devoted to study of the composition of tobacco leaf of various types and grades, and 3.0 to chemical composition of tobacco smoke, primarily cigarette smoke. In addition, the Cigar Manufacturers' Association of America supports parallel effort at Wyndmoor equivalent to two professional man-years; this is a study of cigar smoke.

The Department sponsors research, equivalent to about 0.7 professional man-year, under a P. L. 480 grant to the University of Sao Paulo, Brazil. This is a study of the changes which occur in the composition of tobacco leaves during curing and fermentation.

RELATED PROGRAMS OF STATE EXPERIMENT STATIONS AND INDUSTRY

State Experiment Stations in 1961 reported a total of 5.1 professional man-years, divided as follows: chemical composition and physical properties, 3.4 professional man years; chemical composition of tobacco smoke, 0.1, and new and improved processing technology, 1.6.

State stations in the Northeastern and Southern Regions are engaged in this work.

Industry and other organizations, including tobacco companies and chemical companies, conduct and support extensive research. Tobacco companies' expenditures for in-house research are equivalent to about 200 professional man-years. This effort is directed to improvements in quality and reduction in costs. In the programs, the analysis of leaf and smoke to correlate composition and to compare competitive products, and the improvement of procedures to determine constituents, receive considerable attention. New flavoring agents are investigated, and new methods investigated for producing "homogenized tobacco leaf" or "sheet tobacco" for cigarettes, or cigar binders and wrappers. Through grants of various types, tobacco companies support a research effort equivalent to about 40 professional man-years. Most of this research is devoted to the biological effects of smoking.

Chemical companies conduct an in-house research effort equivalent to about 25 professional man-years. This work includes development of new tobacco flavoring agents, "sheet toabcco" processes, and new and improved machinery for tobacco manufacturing processes.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical and physical properties of tobacco leaf.

In the course of a study of the composition of the resinous substances extracted from flue-cured leaves with hexane and ethanol, the following previously unreported substances were isolated: beta-sitosteryl glucoside, gamma-sitosterol, stigmasterol, ergosterol, an unknown steroidal glycoside, campesterol, alkyl phthalates, isoeugenol, cycloparaffins and a polyene. A study of the amounts of free higher fatty acids in various types and grades was carried out; in flue-cured tobacco, leaves of higher quality tended to have larger amounts of linolenic acid. A method was developed for determining "solanesol-like substances" (SLS) in tobacco; SLS consists mostly of solanesol, plus small amounts of its oxidation products. The levels of SLS (0.4-2.5% of leaf weight) vary with tobacco type but not with quality grade; the levels found in this work are much higher than had been previously believed.

B. Chemical composition of smoke.

Work by fellows supported by the Cigar Manufacturers' Association of America established that the principal volatile acids of cigar smoke are formic and acetic acids, which account for 75% of the volatile acids. There is evidence for propionic through n-heptylic acids in the remaining 25%. Non-volatile acids identified in cigar smoke

include succinic, furoic, lactic, oxalic, glycolic, levulinic, benzoic, malic, phthalic, glutaric, adipic and palmitic.

The acid content of cigar smoke is less than that of cigarette smoke.

A survey of the neutral components of cigar smoke indicated the presence of aliphatic paraffins, silicones, formaldehyde, acetaldehyde, propanal, normal-butanal, methyl ethyl ketone and acetone.

Study of the composition of cigarette smoke has been started.

PUBLICATIONS REPORTING RESULTS OF USDA AND COOPERATIVE RESEARCH

Chemical and physical properties of tobacco leaf.

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Senn, T. L., Griffith, R. B., Ogg, C. L., and Pernell, R. T. 1961. Alkaloid studies of acceptable and discount varieties of flue-cured tobacco grown in 1957. U. S. Dept. Agr., Agr. Research Service Circ. ARS-73-24, 36 pp.

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Chemical composition of smoke.

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Schepartz, A. I. 1961. Paper chromatography of the 2,4-dinitrophenylhydrazones. Extension of the Huelin method. J. Chromatog., 6, pp. 185-186.

Schmeltz, I. and Schlotzhauer, W. S. 1961. Volatile acids of cigar smoke. Tobacco Science No. 5, pp. 92-94.

AREA NO. 10. MAPLE SAP AND SIRUP - PROCESSING AND PRODUCTS

Problem. The extensive unused stands of sugar maple are largely in infertile and hilly areas of marginal value to agriculture, areas commonly devoted to small scale dairy farming. Under proper circumstances, maple sirup could be a seasonal crop of value equal to or exceeding that of the other farm products. By developing proper means of sap handling and processing, by maintaining high quality in the sirup, and by developing new outlets for all maple products, not only can these marginal farms be greatly benefited but the existing maple industry in 14 states put on a higher economic plane.

USDA PROGRAM

The Department has a continuing program involving chemists, biochemists and microbiologists engaged in both basic studies and the application of known principles to the solution of problems affecting maple sirup producers. Most of this work is conducted at Wyndmoor, Pa. Contract research with Michigan State University at East Lansing, Mich. on controlling microbial activity in maple sap with paraformaldehyde has been terminated. Contract research on factors affecting the quality of maple sirup at the Ohio Agricultural Experiment Station, Wooster, Ohio, continues.

The Federal scientific effort devoted to research in this area totals 7.5 professional man-years. Of this number, 2.9 are devoted to study of the chemical composition and physical properties of maple sap and sirup; including 0.3 under the Wooster contract; 2.2 to microbiology of maple products including 0.2 under the East Lansing contract; 1.4 to study of new and improved food products and processing technology; and 1.0 to high-flavored maple products.

In the research work cooperation is maintained with personnel of the Federal Extension Service in maple producing states, and with Cornell University.

RELATED PROGRAMS OF STATE EXPERIMENT STATIONS AND INDUSTRY

State Experiment Stations in 1961 reported a total of 0.5 professional man-year; 0.3 to chemical composition and physical properties and 0.2 to microbiology of maple sap and sirup; state stations of the North Central region are conducting this work.

Industry and other organizations, including maple orchard owners, are conducting a valuable research program on maple sap and sirup totaling about 14 professional man-years. Sirup and food processing companies carry on applied research (about 5 professional man-years) on the intensification of maple flavor and the formulation of high-flavor

products in blended cane-maple table sirups. They are also conducting research on tests for the detection of adulterated maple sirup; and on methods for packing and handling bulk sirup.

Equipment manufacturers are engaged (about 4 professional man-years) in the development of plastic tubing and fittings, and of manufacturing processes for pelleting, packaging and dispensing germicides and sanitizing chemicals developed by the Department for use in sap harvesting. Evaporators are being redesigned to permit substitution of oil burners for wood fuel.

Sap producers and their associations in six states are doing research (about 5 professional man-years), often in cooperation with the Department and State stations, on the correlation of tree spacing, size, soil types, weather conditions and effect of disease and insects, with sap quality and yield, and on the development of improved systems for installing plastic tubing for sap collection.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties.

1. Flavor Components of Maple Sirup. The flavor fraction of maple sirup has been separated into four major and several minor components. Three of the major components are syringaldehyde and vanillin (both previously reported) and probably coniferyl alcohol; the fourth component is not yet identified. Chloroform extracts of sap do not contain either syringaldehyde or vanillin, hence these are evidently formed during "boiling down." Maple sap does contain lignin and since maple tree lignin is the syringal type, lignin may be a precursor of at least some flavor components.

Progress has been made in identifying the components responsible for the flavor of "buddy" (i.e., late-in-the-season) sirup. In "buddy" sap the nitrogen content is several hundredfold greater than in normal sap, and is attributed to a build-up of amino acids normally found in plant juices. In normal sap, on the other hand, one compound, not yet identified, accounts for 80% of the amino fraction.

2. Color Components of Maple Sirup. The substances responsible for the color of maple sirup occur in very low concentrations (10-20 parts per million) but a dextran ion exchange column has made possible their isolation and concentration. Qualitative chemical tests show that the colorant contains carbonyl, carboxyl, reducing and hydroxyl groups; phenolic and proteinaceous groups are absent. These tests indicate that the colorant is composed of sugar and acid fragments, which are probably also flavor components, and that the colorants differ from the flavor bodies in that they contain no phenolic groups.

3. Maple Sugar Sand. Good progress has been made in elucidating the correlation of the formation and composition of sugar sand (the organic acid salts of calcium and magnesium that precipitate during evaporation of sap to sirup) with soil types and other factors. Statistical analysis of the data showed that the least amount of sugar sand was produced in bushes with southern exposures, and the greatest amount in bushes with northern exposures and highest elevations. There was no apparent relationship between soil types and amounts of sugar sand produced. Amounts of sugar sand increased as the season progressed.

B. Microbiology.

1. Improved Maple Products Through Microbial Fermentation. The incubation of freshly-harvested maple sap with a strain of Pseudomonas geniculata for 24 hours at 23° C. modified the sap so that it produced a maple sirup with intensified maple flavor. When "buddy" maple sap was inoculated with a Pseudomonas strain under similar conditions, and processed to sirup, the resulting sirup had the characteristic maple flavor and there was no trace of "buddiness." Since "buddy" sirup cannot be used as table sirup under State and Federal regulations, this discovery is highly significant to the industry.

Additional insight into the fermentation has been obtained. The limiting factor for growth is available nitrogen; the organism metabolizes malic acid; one product of fermentation is butyric acid; under certain conditions of temperature and concentration of organisms, 5 parts per million of formaldehyde does not inhibit growth.

2. Prevention of Microbial Growth in the Taphole.

During this report period, the use of paraformaldehyde pellets as tap hole germicides was tested successfully under commercial conditions: all sirup was of top quality and yields were increased 50-100 percent. The Food and Drug Administration, in a regulation published in the Federal Register of February 20, 1962, permits the use of paraformaldehyde in tapholes to prevent microbial and fungus growth, provided the formaldehyde content of the resulting sirup does not exceed two parts per million. It is estimated that the paraformaldehyde development will be worth at least $1 million annually to producers.

C. New and Improved Food Products and Processing Technology; High-flavored Maple Products.

In this report period work was confined to preliminary experiments to apply some findings under composition and microbiological studies.

PUBLICATIONS REPORTING RESULTS OF USDA AND COOPERATIVE RESEARCH

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New and Improved Food Products and Processing Technology; High-flavored Maple Products

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AREA NO. 11. HONEY - PROCESSING AND PRODUCTS

Problem. Essential pollination of over fifty crops depends almost completely on the honeybee because of changes in agricultural practices over recent years. The importance of the beekeeper in our economy is thus emphasized. As most of his income results from the sale of honey, the beekeeper not only is subject to uncertainties of crop and weather, but must also contend with disease, losses of crop and bees from insecticides, rising costs of needed equipment and materials, lack of trained help, all compounded with uncertain and depressed markets for honey. Because of the relatively small size of operations and the scattered nature of the industry, the honey producer is out-researched, out-promoted and out-advertised by competing sweetening agents. Improved processing methods and equipment, better control of product quality, outlets for lower-grade honey, stable export markets, increased use of honey, both in food manufacture and the home, and increased industrial use of byproducts are all needed to provide an expanding market and encourage the beekeeping industry.

USDA PROGRAM

The Department has a continuing long term program involving chemists and biochemists engaged in both basic studies and the application of known methods and principles to the solution of honey producers' problems. Most of this work is done at Wyndmoor, Pa.; contract research on the evaluation of honey for selected biological properties has also gone forward at Tucson, Arizona, by the University of Arizona.

The Federal scientific effort devoted to research in this area totals 6.2 professional man-years. Of this, 2.0 are devoted to chemical composition and physical properties, 3.6 to enzymes, 0.4 to new and improved food products and processing technology, and 0.2 to the contract research.

RELATED PROGRAMS OF STATE EXPERIMENT STATIONS AND INDUSTRY

State Experiment Stations in 1961 reported a total of 0.7 professional man-year divided as follows: 0.3 to chemical composition and physical properties, 0.3 to new and improved food products and processing technology, and 0.1 to new and improved pharmaceutical and biological uses. State stations in the North Central, Northeastern, Southern and Western regions are engaged in this work.

Industry and other organizations, including honey producers and packers conduct or support a small research program, totaling about 1.5 professional man-years. This program is devoted to improving processing procedures and equipment, developing new products, improving tests for the purity of beeswax, and conducting a small nutrition study.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Chemical Composition and Physical Properties.

The comprehensive analytical study of more than 500 honey samples, representing 83 single floral types and 93 blends of known composition, has been completed and the results published. The publication, Technical Bulletin 1261, will doubtless become the standard reference in the field. In this study the following characteristics of each sample were determined: moisture, color, tendency to granulate, carbohydrates (dextrose, levulose, sucrose, maltose, higher sugars), acidity (pH and free acid), lactone, ash, nitrogen content and diastase value. This is the most complete evaluation ever made on domestic honeys.

B. Enzymes.

Sensitive methods have been developed for determining sucrase, maltase and amylase (diastase) activity, and there is evidence that some of these enzymes exist in multiple forms. The diastase content of honey has long been used in Europe (an important market) as a measure of the heating to which a honey has been exposed; however, it has now been established for the first time that over a storage period of 12 to 18 months, without heating, a honey may lose enough diastase to fall below the minimum values required for European acceptance as table honey.

C. New and Improved Products and Technology; Biological Properties.

In the contract study of biological properties, some previously reported activities were confirmed; these include a bactericidal effect, stimulation of the growth of yeast, and stimulation of appetite in rats. Inconclusive results were obtained for plant-root stimulation, and negative results were noted for estrogenic activity and alleviation of wrist stiffness in guinea pigs. This work has been completed.

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AREA NO. 12 - REPLACEMENT CROPS - UTILIZATION POTENTIAL - EASTERN REGION

Problem. Farmers could achieve economic use of their land if new and profitable crops were available that would have different end uses than crops presently grown. For example, it would be advantageous to develop a new oilseed crop yielding unique fatty acids that could find industrial use in applications for which acids from presently available domestic oilseed crops are unsuitable.

To develop a new crop, three basic steps are involved (1) survey of wild plants, in cooperation with plant scientists, to identify those having both potentially valuable components and promising agronomic potential for use in the U.S.; (2) detailed physical and chemical characterization of components of interest to obtain clues to likely end uses; (3) selection of the most promising species, followed by additional utilization research to explore uses and demonstrate industrial potential and by additional agronomic research to establish proper cultural practices and select the best strains and varieties.

Only after these steps have been successfully accomplished can a proposed new crop be offered to agriculture and industry for introduction and development. Obviously, a program of this type is a long-range one. Yet such long-range research is necessary if agriculture and the nation are to benefit from availability of the best practical crop plants.

To achieve this objective, survey and characterization work needs to be greatly increased, since the greater the number of species examined, the greater will be the opportunities for finding plants meeting the criteria of high utilization and agronomic potential. Work of the Department has already revealed several promising sources of new potentially valuable water soluble gums, pulp fibers, and oils containing unique fatty acids such as hydroxyunsaturated acids, capric acid,epoxy acids, and unusual long-chain fatty acids. In order to demonstrate the potential of these new materials, further work is required on their physical and chemical properties and reactions, on processing to obtain maximum recovery from source plants and on byproducts from processing, such as oilseed meals.

USDA PROGRAM

The Department has a continuing program involving chemists engaged in both basic and applied studies directed to the development of profitable new crops.

At Wyndmoor, Pennsylvania, work goes forward on the assay of Canaigre tubers for their tannin content and of Dioscorea tubers for their content of steroidal sapogenins, which are intermediates in the synthesis of corticoid drugs. This work is cooperative with the Crops Research Division.

Also at Wyndmoor, research work is carried out on the utilization of oil containing epoxy fatty acids, from the seed of Indian ironweed (Vernonia anthelmintica). This work is cooperative with the Northern Utilization Research and Development Division, the Crops Research Division and the Western Utilization Research and Development Division. The Federal scientific effort at Wyndmoor Totals 5.0 professional man-years. Of this number, 2.0 are devoted to composition of Dioscorea and Canaigre, and 3.0 to utilization of oilseeds containing epoxidized oils.

During this report period, work on the analysis of spices, that had been conducted by fellows supported by the American Spice Trade Association, was terminated.

Related programs of all State Experiment Stations, and industry and other organizations is reported by the Northern Utilization Research and Development Division in that Division's Summary of Current Program and Preliminary Report of Progress.

REPORT OF PROGRESS FOR USDA AND COOPERATIVE PROGRAMS

A. Assay of Dioscorea and Canaigre.

In this reporting period, assays of 661 agronomic samples of Dioscorea were carried out, substantially all of them Dioscorea spiculiflora, which is the species of choice as a potential replacement crop because of its gentrogenin content. Gentrogenin is a good starting point for the synthesis of many drugs. The assay results were reported to the Crops Research Division for consolidation with the agronomic studies of that Division.

Analysis of 225 samples of canaigre tubers from the 1961 crop, together with analysis of earlier samples, have established the existence of strains which contain over 40% tannin and which yield extracts with purities over 70. This work has established the usefulness of canaigre as a domestic source of vegetable tannin--necessary in the manufacture of heavy leather--but at present prices canaigre cannot compete with imported quebracho. Hence the most promising strains are being maintained in germ plasm banks; three new planting sites have been selected in the Tonto National Forest, and 3 high-tannin, high-yielding varieties were planted in quantity to permit rapid expansion in case of emergency. The germ plasm banks also include 183 selected strains that have potential for future breeding or propagation studies.

B. Utilization of Oilseeds Containing Epoxidized Oils.

The seed of Indian ironweed (Vernonia anthelmintica Willd.) contains 25% of a unique oil, which is rich in esters--principally the triglyceride--of vernolic acid (Epoxyoleic acid, $CH_3(CH_2)_4$ - $\underset{O}{CHCHCH_2}CH{=}CH(CH_2)_7COOH$). These esters represent 70-75% of the oil, expressed as the triglyceride. The oil is easily obtained from the seed by solvent extraction; when the extract is cooled to low temperatures, trivernolin can be recovered in good yield (60% of the oil) and high purity (95%).

The unmodified oil, trivernolin, vernolic acid and the barium and cadmium salts of vernolic acid all stabilize polyvinyl chloride against heat and light. Methods have been developed for the preparation of methyl vernolate in good yield and high purity by transmethylation of either the oil or pure trivernolin. This ester should also be a good stabilizer for vinyl plastics.

The oilseed contains enzyme systems which bring about chemical reactions in the oil. Preliminary experiments indicate that the activity of these enzymes may be controlled to produce free vernolic acid, 1,3-divernolin and (plus) threo-12,13-dihydroxyoleic acid.

This work was described at scientific meetings and several papers are now in the process of publication.

C. Analysis of Spices and Spice Oils.

Under the fellowships supported by the American Spice Trade Association, an analysis system was devised, employing gas chromatography and infrared spectrophotometry, for detecting in spice oils components. that are characteristic of the geographical origin. Three characterizing components were identified for black pepper, four for nutmeg, four for ginger and two for cassia. The ratios of gas chromatogram peak heights of these components were characteristic of the geographical origin of the spice.

This work has now been terminated.

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Line Project Check List -- Reporting Period July 1, 1961 to June 30, 1962

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Subheading
E6 2	Milk products utilization investigations. Program Leadership	Wyndmoor, Pa. & Washington, D.C.		
E6 2-38 (Rev.)*	Characteristics of the casein complex in milk	Washington, D. C.	Yes	1,2-B-2
E6 2-64 (C)	Improving the stability of butter flavor	Ames, Iowa	Yes	1,2-C
E6 2-68	The chemistry of bacterial spores	Washington, D. C.	Yes	1,2-E
E6 2-70	Development of new outlets for butterfat	Washington, D. C.	Yes	1,2-L
E6 2-71	Processing high nonfat solids-low fat milk	Washington, D. C.	No.	
E6 2-73*	Physical-chemical studies of dry whole milk	Washington, D. C.	Yes	2-H-5
E6 2-74	Chemistry of flavor changes during processing and storage of dry milk	Washington, D. C.	Yes	1,2-C
E6 2-75	The development of a commercially feasible process for preparing readily dispersible dry whole milk of good flavor and adequate shelf life	Wyndmoor, Pa.	Yes	1,2-H-1; H-3
E6 2-77	Improvement in the quality of Cheddar cheese	Washington, D. C.	Yes	1,2-J
E6 2-79	Factors affecting flavor score and storage stability of foam-dried whole milk	Washington, D. C.	Yes	1,2-H-1; H-2; H-4
E6 2-81	Removal of radioactive contamination from milk	Beltsville, Md.	Yes	1,2-M
E6 2-82 (C)	The chemical nature of stale flavor in aged sterile milk	Urbana, Ill.	Yes	1,2-C; K
E6 2-83 (C)	Chemistry of Cheddar cheese flavor	Columbus, Ohio	Yes	1,2-C; J
E6 2-84	A laboratory and pilot plant study of the effect of chemical additives on the storage stability of evaporated milk	Washington, D. C.	Yes	1,2-K
E6 2-85	Interactions of milk proteins in solution	Wyndmoor, Pa.	Yes	1,2-A; B-1
E6 2-87**	Methods for making fat-free and low-fat cheese	Washington, D. C.	Yes	1,2-J
E6 2-88**	Physico-chemical studies of factors influencing milkfat/plasma emulsion stability	Washington, D. C.	Yes	1,2-B-2; K
E6 2-89 (C)**	Development of improved techniques for evaluating importance of flavors in new concentrated milk products	Corvallis, Ore.	Yes	1,2-C
E6 2-90**	Improvement of concentrated whole milk products	Washington, D. C.	Yes	1,2-N
E6 2-91**	Properties of components of milk related to physical changes during processing and storage	Wyndmoor, Pa.	Yes	1,2-B-1; B-2,F
E6 2-92**	Development of increased food outlets for nonfat milk solids	Washington, D. C.	No	
EU P1	Pioneering Research Laboratory, Allergens in Agricultural Products	Washington, D. C.	Yes	1,2-B-1; G
EU P2	Pioneering Research Laboratory, Animal Proteins in Agricultural Products	Wyndmoor, Pa.	Yes	1,2-B-1; F

* Discontinued during report year.
** Initiated during report year.

Line Project Check List -- Reporting Period July 1, 1961 to June 30, 1962

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj.Incl. in Summary of Progress	Line Proj.Incl. in Area & Subheading
UR-A7-(60)-5	Milk coagulating enzymes	India		
UR-A7-(60)-13	Buffalo milk in cheese manufacturing	India		
UR-A7-(60)-22	Proteose-peptone fraction of milk	India		
UR-E8-(60)-1	Growth-promoting factors for lactic acid bacteria	Finland		
UR-E8-(60)-5	Effect of feed components on milk and milk products	Finland		
UR-E9-(60)-46	Non-protein nitrogenous constituents of milk	France		
UR-E9 (60)-47	Proteolytic activity of rennin on casein	France		
UR-E15-(60)-16	Food products from milk and fruit concentrates	Italy		
UR-E21-(60)-7	Increasing vitamin B in cheese	Poland		
UR-E25-(60)-18	Protein destabilization in frozen milk	Spain		
UR-E29-(60)-31	Microorganisms in dairy products	United Kingdom		
UR-E29-(60)-41	Studies on selected enzymes of milk	United Kingdom		
UR-S3-(60)-10	Proteolytic enzyme activity in milk	Brazil		
UR-E9-(10,60)-80	Structure of nucleic acids	France		

Line Project Check List -- Reporting Period July 1, 1961 to June 30, 1962

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Sub-heading
E6 5	Meat utilization investigations. Program Leadership	Wyndmoor, Pa. and Washington, D. C.		
E6 5-7*	Fractionation of the fibrillar protein complexes of meat	Beltsville, Md.	Yes	3-A
E6 5-9 (C)	A study of the relationship of dry milk solids used as binders to product quality and processing characteristics in meat food products	East Lansing, Mich.	Yes	3-C
E6 5-10*	Development of improved methods of curing meat through studies of salt-tolerant micro-organisms and their relation to cured meat quality	Beltsville, Md.	Yes	3-E
E6 5-13*	Improving the quality of meat through studies of low-temperature tolerant bacteria	Beltsville, Md.	Yes	3-E
E6 5-15 (C)	Tenderness reversion in frozen beef as affected by holding temperatures and freezing techniques	Stillwater, Okla.	Yes	3-F
E6 5-17	Studies of chemical and microbiological factors involved in the freezing of meats	Beltsville, Md.	Yes	3-B; 3-E
E6 5-18 (C)	A study of meat pigments and their relationship to lipid oxidation and stability of frozen, cured, and uncured meats	Tallahassee, Fla.	Yes	3-B
E6 5-19	Identification of substances responsible for flavor and aroma in meat	Beltsville, Md.	Yes	3-D
E6 5-20	Chemical reactions involved in meat curing	Beltsville, Md.	Yes	3-C
UR-E8-(60)-4	Effect of microorganisms on sausage flavor	Finland		
UR-E15-(60)-13	Studies on beef canning	Italy		
UR-E29-(60)-15	Enzymes attacking animal connective tissue	United Kingdom		
UR-E29-(60)-18	Accelerated freeze-drying of meat	United Kingdom		
UR-E29-(60)-24	Biochemical properties of pork muscle	United Kingdom		

* Discontinued during report period

Line Project Check List -- Reporting Year July 1, 1961 to June 30, 1962

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Sub-heading
E6 3	Animal Fats and Oils and Special Products Utilization Investigations	Wyndmoor, Pa.		
E6 3-44*	Fractionation and analysis of lipids	Wyndmoor, Pa.	Yes	4-A-1
E6 3-46**	Plastics from animal fats. 1. Copolymers octadecyl acrylate	Wyndmoor, Pa.	Yes	4-B-2
E6 3-49**	Polymers containing fatty epoxides	Wyndmoor, Pa.	Yes	4-B-2
E6 3-52****	Synthesis of pure individual mixed triglycerides	Wyndmoor, Pa.	Yes	4-A-3
E6 3-53	Addition of nucleophiles to fatty derivatives	Wyndmoor, Pa.	Yes	4-D-1
E6 3-55	Epoxidized and hydroxylated fat derivatives for industrial use	Wyndmoor, Pa.	Yes	4-D-3
E6 3-56	Phosphorus, sulfur, oxygen and nitrogen-containing fat derivatives	Wyndmoor, Pa.	Yes	4-D-4
E6 3-57	Autoxidation of fatty substances in emulsions	Wyndmoor, Pa.	Yes	4-A-2
E6 3-58	Soap-detergent combinations based on animal fats	Wyndmoor, Pa.	Yes	4-C-2
E6 3-59 (C)	Determination of the structural characteristics of triglycerides	Villanova, Pa.	Yes	4-A-3
E6 3-60 (C)	Dielectric properties of fatty peroxides	Philadelphia, Pa.	Yes	4-D-5
E6 3-61 (C)	Substituted vinyl monomers and polymers from animal fats	Tucson, Ariz.	Yes	4-B-1
E6 3-62	Development of synthetic lubricants from animal fats	Wyndmoor, Pa.	Yes	4-B-3
E6 3-63	Organic-inorganic compounds from fats	Wyndmoor, Pa.	Yes	4-B-2
E6 3-64***	Structure of components and derivatives of animal fats	Wyndmoor, Pa.	Yes	4-A-3
E6 3-65***	Fractionation and analysis of lipids and oxidation products	Wyndmoor, Pa.	No	---
E6 3-66***	Polymerizable amides from animal fats	Wyndmoor, Pa.	Yes	4-B-2
E6 3-67 (C)***	Structural characteristics of organic peroxides	Pittsburgh, Pa.	No	

* Superseded by E6 3-65.
** Discontinued during report year.
*** Initiated during report year.
**** Superseded by E6 3-64.

Line Project Check List -- Reporting Year July 1, 1961 to June 30, 1962

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Project Incl. in Summary of Progress (Yes-No)	Line Project Incl. in Area & Subheading
E6 4	Hides, Skins and Leather Utilization Investigations. Program Leadership	Wyndmoor, Pa.		
E6 4-22*	Distribution and properties of hide components	Wyndmoor, Pa.	Yes	5-A-1 5-B-1
E6 4-23*	Improved enzyme-unhairing treatment for hides	Wyndmoor, Pa.	Yes	5-D-2 5-E-1
E6 4-24 (C)*	Composition of basement membrane of cattlehides	Lowell, Mass.	Yes	5-A-2
E6 4-25 (C)	Evaluation of enzymic processes for unhairing hides	Cincinnati, Ohio	Yes	5-D-2 5-E-2
E6 4-26	Reversible shrinkage in leather	Wyndmoor, Pa.	Yes	5-A-3
E6 4-27*	Leathers having increased resistance to deterioration	Wyndmoor, Pa.	Yes	5-D-1
E6 4-28 (C)	Conversion of dialdehyde-tanned skins to salable light leather	Lowell, Mass.	Yes	5-C-2
E6 4-29	Improved water repellency for leather	Wyndmoor, Pa.	Yes	5-C-1
E6 4-30	Modification of hides and skins with dialdehydes	Wyndmoor, Pa.	Yes	5-C-2
E6 4-31 (C)	Design and construction of a dynamic mechanical tester for hides	Philadelphia, Pa.	Yes	5-A-4
E6 4-32	Composition and properties of animal protein residues	Wyndmoor, Pa.	Yes	5-F-1
E6 4-33 (C)	Evaluation of collagen fibers from enzyme-unhaired and lime unhaired hides	Kansas City, Mo.	Yes	5-B-1
E6 4-34	Processes for making commercial quality leather from enzyme-unhaired hides	Wyndmoor, Pa.	No	
E6 4-35	New tanning processes for producing leathers of superior durability	Wyndmoor, Pa.	Yes	5-D-3
E6 4-36	Effect of electrolytes and lipid components on hide properties	Wyndmoor, Pa.	Yes	5-B-1
E6 4-37 (C)**	Noncollagenous proteins of cattlehides	Cincinnati, Ohio	No	
UR-A7-(60)-17	Reaction of polyphenolic tanning compounds with hide proteins (collagen)	Madras, India		
UR-A7-(60)-18	Interrelation of hide quality with rate of tanning and efficiency of tanning	Madras, India		
UR-E8-(60)-3	Fractionation of gelatin and collagen	Turku, Finland		
UR-E15-(60)-5	Microbial damage to exported U. S. hides	Naples, Italy		
UR-E15-(60)-7	Tanning studies on American hides	Naples, Italy		
UR-E29-(60)-2	Deterioration of leather by sweat and heat	Surrey, England		

* Discontinued during report year.
** Initiated during report year.

Line Project Check List - Reporting Year July 1, 1961 to June 30, 1962

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Summary of Progress	Incl. in Area & Sub-heading
E3 6	Potato and other vegetable utilizations - Eastern Region. Program Leadership	Wyndmoor, Pa.		
E3 6-27*	Chemical factors in potato after-cooking discoloration	Wyndmoor, Pa.	Yes	6-A-2
E3 6-31*	Technological research related to potato processing	Wyndmoor, Pa.	Yes	6-C-2
E3 6-37	Development of "instantized" dehydrated potato pieces	Wyndmoor, Pa.	Yes	6-B-1
E3 6-39*	Development of methods of producing potato flakes	Wyndmoor, Pa.	Yes	6-B-2
E3 6-42	Nitrogenous constituents as quality factors in potato processing	Wyndmoor, Pa.	Yes	6-A-1
E3 6-43**	Improvement in storage stability of potato flakes and flakelets	Wyndmoor, Pa.	Yes	6-C-1

* Discontinued during report year
** Initiated during report year

Line Project Check List - Reporting Year July 1, 1961 to June 30, 1962

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Sub-heading
E3 6	Potato and other vegetable utilizations - Eastern Region. Program Leadership	Wyndmoor, Pa.		
E3 6-36(C)*	Evaluation of the quality improvement in eastern canned vegetables through use of high-temperature short-time processing	New Brunswick, N.J.	Yes	7-C-1
E3 6-38	Studies on plants containing inhibitors for pectinolytic and celluloytic enzymes	Wyndmoor, Pa.	Yes	7-A-1
E3 6-41	Development of new types of dehydrated vegetables through modification of internal structure	Wyndmoor, Pa.	Yes	7-B-1

* Discontinued during report year

Line Project Check List -- Reporting Year July 1, 1961 to June 30, 1962

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Project Incl. in Summary of Progress	Line Project Incl. in Area & Sub-heading
E3 3	Apples and Other Fruit Utilization Investigations - Eastern Region. Program Leadership.	Wyndmoor, Pa.		
E3 3-27*(C)	Utilizing fruit essences and concentrates in frozen desserts	College Park,Md.	Yes	8-B-4
E3 3-29*(C)	Relation of pectin, cellulose and other carbohydrates to texture of processed apple products	College Park,Md.	Yes	8-A-2
E3 3-30	Development of improved apple cider	Wyndmoor, Pa.	Yes	8-B-2
E3 3-31*	Quality improvements in dehydrated fruit products	Wyndmoor, Pa.	Yes	8-B-3
E3 3-32	Rapidly-reconstitutable dried fruit products	Wyndmoor, Pa.	Yes	8-B-1
E3 3-33**(C)	Relation of physical and chemical properties to processing characteristics of eastern peaches	New Brunswick,N.J.	Yes	8-C-1
E3 3-34**	Improvement of processed cherries through studies on composition and post-harvest treatments	Wyndmoor,Pa.	Yes	8-A-1
E3 3-35***(C)	Relation of apple cell wall constituents to textural quality of processed products		No	

* Discontinued during report year.
** Initiated during report year.
*** Initiated during report year. Contract planned for execution in F.Y. 1963, with Univ. of Maryland, College Park, Md.

Line Project Check List -- Reporting Year July 1, 1961 to June 30, 1962

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Inc. in Summary of Progress	Line Proj. Inc. in Area & Sub-heading
E5-3	Tobacco Investigations. Program Leadership	Wyndmoor, Pa.		
E5 3-2 (Rev.)*	Chemical composition of cigar tobacco smoke	Wyndmoor, Pa.	Yes	9-B
E5 3-4**	Composition of the resins and polyphenols of tobacco	Wyndmoor, Pa.	Yes	9-A
E5 3-5***	Acids and bases of cigar smoke	Wyndmoor, Pa.	Yes	9-B
E5 3-6***	Composition of cigarette smoke	Wyndmoor, Pa.	Yes	9-B
E5 3-7***	Composition of oxidation products and related substances of tobacco	Wyndmoor, Pa.	Yes	9-A
UR-S3-(50)-4	Fermentation studies on tobacco	Brazil		

* Superseded during report year by E5 3-5.
** Discontinued during report year.
*** Initiated during report year.

Line Project Check List -- Reporting Year July 1, 1961 to June 30, 1962

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Project Incl. in Summary of Progress	Line Project Incl. in Area & Sub-heading
E5 1	Sugars and Sirups Investigations. Program Leadership.			
E5 1-60	Microbial fermentation of sap and selective screening for organisms that impart desirable characteristics	Wyndmoor, Pa.	Yes	10-B-1
E5 1-63(C)*	Elimination of contamination by micro-organisms through use of trioxymethylene paraformaldehyde	East Lansing, Mich.	Yes	10-B-2
E5 1-67(C)	Improvement in processing and quality by study of factors affecting sugar sand formation	Wooster, Ohio	Yes	10-A-3
E5 1-74	Chemical compounds responsible for maple flavor; precursors of maple flavor	Wyndmoor, Pa.	Yes	10-A-1

* Discontinued during report year

Line Project Check List -- Reporting Year July 1, 1961 to June 30, 1962

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Project Incl. in Summary of Progress	Line Project Incl. in Area & Sub-heading
E5 1	Sugar and Sirups Investigations. Program Leadership	Wyndmoor, Pa.		
E5 1-62(C)	Investigation of the biological activities of honey	Tucson, Ariz.	Yes	11-C
E5 1-68	Isolation, characterization and properties of honey enzymes	Wyndmoor, Pa.	Yes	11-B

Line Project Check List -- Reporting Year July 1, 1961 to June 30, 1962

Work & Line Project Number	Work and Line Project Titles	Work Locations During Past Year	Line Proj. Incl. in Summary of Progress	Line Proj. Incl. in Area & Sub-heading
E5 5	New and Replacement Crops Utilization Investigations Program Leadership	Wyndmoor, Pa.		
E5 5-31	Development of plant sources of precursors for steroid hormones: Assay of agronomic samples for steroidal sapogenins	Wyndmoor, Pa.	Yes	12-A
E5 5-35*	Identification and determination of the important chemical components of spices and spice products	Wyndmoor, Pa.	Yes	12-C
E5 5-39	New crop seed epoxy-containing oils	Wyndmoor, Pa.	Yes	12-B
E5 5-43	Development of canaigre as a source of tannin	Wyndmoor, Pa.	Yes	12-A

* Discontinued during report year